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SYNTHESIS AND STRUCTURE OF A NEW CLASS OF METALLOPHTHALOCYANATO--ETC(U)
JAN 80 M TSUTSUI, S OMIYA   N00014-75-C-0417
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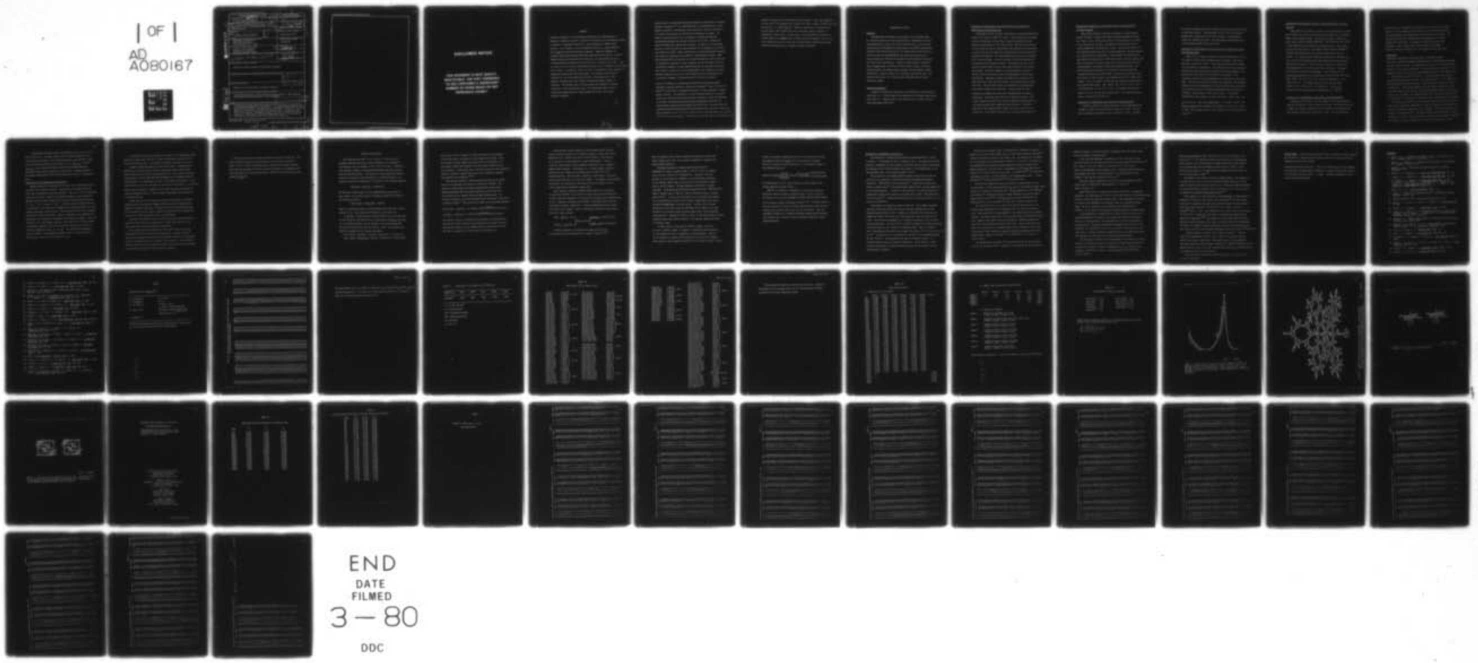

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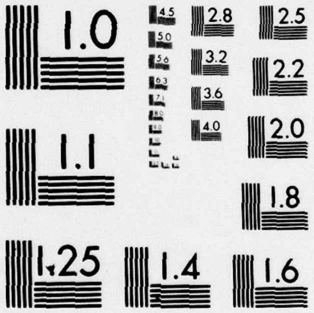
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Carbonylphthalocyanato(pyridine)ruthenium(II) PcRu(CO)(Py), carbonylphthalocyanatopyridineosmium(II) PcOs(CO)(Py), and carbonylphthalocyanato (tetrahydrofuran)ruthenium(II) PcRu(CO)(THF) were prepared in pure form. They are among the first reported metallophthalocyanines with a carbonyl as one of their axial ligands. Furthermore, several new ways of synthesizing these complexes in quantitative yields have been established. The structure of PCOs(CO)(Py) elucidated by the X-ray diffraction analysis.

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ABSTRACT

During our synthesis of phthalocyanatoruthenium(II) and phthalocyanatoosmium(II) complexes reported by Berizin and Sennikova, and by Krueger and Kenny we observed a remarkable solubility of these complexes in common organic solvents. The solubility of these complexes enabled us to isolate several pure reported and new phthalocyanine complexes. Among the complexes we studied, carbonylphthalocyanato(pyridine)ruthenium(II) [PcRu(CO)(Py), carbonylphthalocyanatopyridineosmium(II)[PcOs(CO)(Py)], and carbonylphthalocyanato (tetrahydrofuran)ruthenium(II)[PcRu(CO)(THF)] were isolated in pure form. They are among the first reported metallophthalocyanines with a carbonyl as one of their axial ligands. Furthermore, several new ways of synthesizing these complexes in quantitative yields have been established. The structure of PcOs(CO)(Py) elucidated by the X-ray diffraction analysis. The osmium ion is octahedrally coordinated with the carbonyl and pyridine groups axially coordinated. The pyridine ring is tilted slightly with respect to the perpendicular to the phthalocyanine ring. The interplanar angle is 98.6°. Interesting comparisons may be made between PcOs(CO)(Py) and related porphyrin complexes;

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Phthalocyanine and metallophthalocyanines demonstrate significant electrical and photo properties,[1,2] e.g. semiconductivity, photoconductiviety, photochemical reactivity, luminescence, and fluorescence, which are relevant to the current world problem of conversion and production of energy. The number of publications per year on chemistry of phthalocyanines and metallophthalocyanines has tripled during the past decade. During 1977, nearly 700 papers and patents were published. In spite of the increasing interest in this area and the near completion of the synthesis of all metal ions having the normal classic configuration, basic phthalocyanine chemistry has lacked the advantage of any dynamic or extraordinary progress. Many metalloporphyrins[3] have been made and studied extensively, and a large body of coordination chemistry of these complexes has been developed, while the coordination chemistry of metallophthalocyanines has been poorly developed. This contrast is mainly due to the significantly lower solubility of metallophthalocyanines and has led to the presumption that the development of coordination chemistry of phthalocyanines is difficult in general.

During our synthesis of pthalocyanatoruthenium(II) and phthalocyanatoosmium(II) complexes reported by Berizin and Sennikova[4,5] and by Krueger and Kenny,[6a] we have found a remarkable solubility of these complexes in common organic solvents. The solubility of these complexes enabled us to isolate several pure reported and new phthalocyanine complexes. Among the complexes we studied, carbonylphthalocyanato(pyridine)ruthenium(II)[PcRu(CO)(Py)], carbonylphthalocyanato(pyridine)osmium(II)[PcOs(CO)(Py)], and carbonylphthalocyanato(tetrahydrofuran)ruthenium(II)[PcRu(CO)(THF)] were isolated in pure form. These compounds are among the first reported metallophthalocyanines with carbonyl as one of their axial ligands. Furthermore, several new ways of synthesizing these complexes in quantitative yields have been established. Since the completion of this work,[6c] the preparation of PcRu(CO)(L)L (L=Py, 4-MePy; 4-t-BuPy) has been reported by N. P. Farrell *et al.*[6b] However, the methods of preparation described herein, which differ from their techniques, produce products in considerably higher yield. In this paper we also describe the structure of PcOs(CO)(Py)Py as determined from an x-ray diffraction analysis. This is the first reported structure of this type of phtalocyanine complex and provides interesting comparisons with analogous porphyrin complexes.

Experimental Section

Materials

Phthalonitrile (practical) was purchased from the Eastman Kodak Co. and used without any further purifications. Ruthenium-trichloride-(trihydrate)($RuCl_3.3(H_2O)$) and osmium tetraoxide(OsO_4) were purchased from the Ventron Corporation and used without any further purifications. Ruthenium dodecacarbonyl($Ru_3(CO)_{12}$) and osumium dodecrabonyl($Os_3(CO)_{12}$) were purchased from Strem Chemicals and used without purification. Carbon monoxide gas (99.9%) was purchased from the Matheson Gas Company and used without purification. Pyridine (reagent grade), tetrahydrofuran (reagent grade), methylene chloride (spectro grade), chloroform (spectro grade), and benzene (spectro grade) were used without further purification. Neutral alumina (60 - 100 mesh), acid alumina (80 - 200 mesh) and silica gel (60 - 200 mesh) were purchased from the Fisher Scientific Company.

Physical Measurements

Elemental analysis were performed by the Schwarzkopf Microanalytical Laboratory, N.Y. Visible spectra were measured with a Beckman Spectrophotometer Model 24. Infrared spectra were measured with a Beckman Infrared Spectrophotometer Model IR-8.

Isolation of PcRu(CO)(THF) from the Products of the Reaction of $RuCl_3 \cdot 3(H_2O)$ with Phthalonitrile

$RuCl_3 \cdot 3H_2O$ (1.00 g; 3.8 mmol) was heated with excess phthalonitrile (8.00 g; 62.5 mmol) at 250°C for 4 hours. After washing the resulting product with methanol several times, unreacted phthalonitrile was removed by sublimation at 150°C under vacuum. The blue-black residue (4.1 g), which was left at the bottom of the sublimator, was dissolved in aniline (10 mL), and metal-free phthalocyanine was removed from the solution by filtration. The aniline solution was poured into benzene (1.0 L) with stirring. A black precipitate (0.8 g) was separated by filtration from the benzene solution. The solution was then concentrated and dried under vacuum at 100°C until aniline in the solution was removed. The dried residue (3.5 g) was dissolved in benzene (30 mL), and then the solution was chromatographed over silica gel with benzene. Meanwhile, benzene was replaced by chloroform, and a light blue band, which was most intense, was separated from the dark blue band. The light blue eluate was condensed and chromatographed over neutral alumina with chloroform. Another bright blue band followed the original blue band when tetrahydrofuran was added to chloroform. The second blue eluate was condensed and dried. Fine needle crystals (120 mg) with a typical phthalocyanine appearance of red reflection and blue transmission were obtained. The visible spectrum of this compound shows maxima (in nm) at 642 and 581 (in chloroform). The infrared spectrum has an intense peak at 1960 cm^{-1}.

Preparation of PcRu(CO)(Py) from $RuCl_3 \cdot 3(H_2O)$ and Phthalonitrile in Carbon Monoxide

$RuCl_3 \cdot 3(H_2O)$ (100 mg; 0.38 mmol) was heated in molten phthalonitrile (500 mg; 1.9 mmol) under carbon monoxide atmosphere at 250°C for 4 hours. After cooling the product to room temperature, pyridine (5 mL) was added to the cake. The mixture was again heated to the boiling temperature of pyridine for 1 hour. The pyridine solution of the resulting product was cooled to room temperature, and then pyridine was removed first by distillation. Excess amounts of phthalonitrile were removed by sublimation at 150° under vacuum. The blue residue (265 mg) was dissolved in methylene chloride (15 mL), and the solution was chromatographed over neutral alumina with methylene chloride. The first blue band was followed by a second blue band when methylene chloride was replaced with chloroform. The second blue eluate, which was present in large quantity, was condensed and dried at 100°C under vacuum. A fine blue powder (230 mg) with red reflection (PcRu(CO)(Py)) was obtained (84% yield). Anal. Found: Ru, 13.97; N, 17.35. Calc. for PcRu(CO)(Py): Ru, 14.02; N, 17.49. The visible spectrum shows maxima (in nm) at 624 and 581 (in chloroform). The infrared spectrum shows an intense peak at 1965 cm^{-1}.

Preparation of PcRu(CO)(Py) from $Ru_3(CO)_{12}$ and Phthalonitrile

$Ru_3(CO)_{12}$ (100 mg; 0.16 mmol) was added to molten phthalonitrile (500 mg; 1.9 mmol) in air and heated at 250°C for 4 hours. The product was heated with pyridine (5 mL) at 150°C for 1 hour. Pyridine and unreacted phthalonitrile were removed, and the residue was dissolved in methylene chloride. Chromatography of the solution over neutral alumina gave a single blue band with chloroform. The chloroform eluate was condensed and dried. The blue powder (PcRu(CO)(Py)) with red reflection weighed 315 mg (91% yield).

Isolation of PcOs(CO)(THF) from the Products of the Reaction of OsO_4 with Phthalonitrile

OsO_4 (1.0 g; 3.9 mmol) was added to molten phthalonitrile (6.0 g; 46.8 mmol) in a 50 mL flask equipped with a condenser. The mixture was kept at 250°C for 4 hours. The excess phthalonitrile was removed from the product by sublimation. The residue was put in an extraction thimble, and some products were extracted with tetrahydrofuran. The THF solution was condensed and dissolved in chloroform. The chloroform solution was chromatographed over acid alumina with chloroform. The first blue band was collected and condensed. The condensed residue was then chromatographed over neutral alumina with chloroform. The first blue band of the second chromatography was collected and condensed. After drying at 100°C under vacuum, a blue powder (PcOs(CO)(THF)) was obtained (115 mg; 3.7% yield). Anal. Found: Os, 23.20; N, 14.15. Calc. for PcOs(CO)(THF): Os, 23.69; N, 13.96. The visible spectrum of the compound shows maxima (in nm) at 636, and 576 in THF. The infrared spectrum of the compound shows an intense peak at 1930 cm^{-1}.

Preparation of PcOs(CO)(Py) from OsO_4 and Phthalonitrile in Carbon Monoxide

OsO_4 (107 mg; 0.42 mmol) was added to molten phthalonitrile (500 mg; 3.9 mmol) in a 50 mL round bottom flask equipped with a condenser under carbon monoxide stream. The mixture was kept at 250°C for 4 hours under a carbon monoxide atmosphere. The mixture was then cooled to room temperature and dissolved in pyridine (5 mL). The pyridine solution was refluxed for 1 hour under a carbon monoxide atmosphere. Pyridine and phthalonitrile were removed under vacuum from the product. The resulting blue residue was dissolved in chloroform (50 mL), and metal-free phthalocyanine was removed from the chloroform solution by filtration. The chloroform solution was condensed and chromatographed over neutral alumina with chloroform. The single blue band was collected and condensed. A blue powder with red reflection(PcOs(CO)(Py)) was obtained after drying (280 mg; 83% yield). Anal. Found: Os, 22.96; N, 15.48. Calc. for PcOs(CO)(Py): Os, 23.49; N, 15.57. The visible spectrum of the compound shows maxima (in nm) at 632 and 575 (in chloroform). The infrared spectrum of the compound shows an intense peak at 1930 cm^{-1}.

Preparation of PcOs(CO)(Py) from $Os_3(CO)_{12}$ and Phthalonitrile

$Os_3(CO)_{12}$ (100 mg; 0.11 mmol) was added to molten phthalonitrile (500 mg; 3.9 mmol) in a 50 mL round bottom flask. The mixture was kept at 250°C for 4 hours. Pyridine (5 mL) was added to the mixture, and the pyridine solution was refluxed for 1 hour. Pyridine and excess phthalonitrile were removed from the product by distillation and sublimation. The blue residue was then dissolved in chloroform, and metal-free phthalocyanine was separated from the solution by filtration. The chloroform solution was condensed and chromatographed over neutral alumina with chloroform. The single blue band was collected and condensed. After drying at 100°C under vacuum, a blue powder with red reflection (PcOs(CO)(Py)) was obtained (220 mg; 82% yield).

X-ray Study

Crystals of PcOs(CO)(Py) were grown from a chloroform solution. The crystal chosen for intensity measurements was a parallellopiped bounded by {100}, {010} and {001}. The dimensions were 0.20 x 0.20 x 0.09 mm in the direction of a, b and c respectively. It was mounted in a capillary [7] at an arbitrary orientation, but with a approximately parallel to the spindle axis.

Crystal Data are listed in Table 1. An Enraf-Nonius CAD-4 computer controlled diffractometer was used. The radiation (Mo $K\alpha$, $\lambda=0.71069A$) was monochromatized by pyrolitic graphite. The instrument centered the crystal automatically. The setting angles for 25 reflections, measured at + and - 2Θ, were used to index the cell and then were refined to give an orientation matrix, cell constants, and a Niggli matrix[8] which indicated that the system was monoclinic.[9] The systematic absences uniquely determined the space group.

The diffracted intensities were collected using the Θ-2Θ scan technique. Scan speeds, which were determined by a rapid preliminary scan, ranged from 0.28 to 3.35 deg/min. depending on the intensity. Very weak reflections were measured at the maximum rate. The scan range for each reflection was equal to $0.90 + 0.35 \tan \Theta$. Other experimental conditions are described elsewhere.[10] No evidence of crystal decomposition or machine instability was noted.

Independent reflections (4596) were measured out to a sinΘ value of 0.54 or 22.5° in Θ. Of these, 2820 had a net intensity greater than $2\sigma_I$ and were used in analysis. The standard deviation σ_I was defined in terms of the statistical variances of the counts as $\sigma_I{}^2 = \sigma^2_{I(count)} + (0.02I)^2$ $\sigma_{I(count)}$ is the variance determined solely from counting statistics. Structure factors were calculated in the usual way, including correction for partial polarization of the incident beam due to the use of a monochromator.

Determination and Refinement of the Structure

Because there are four molecules in the unit cell of space group $P2_1/n$, all atoms lie in general positions. The position of the osmium atom was found from an unsharpened Patterson synthesis. The rest of the 49 nonhydrogen atoms were found from a series of difference syntheses. Least squares refinement using full matrix methods was carried out minimizing the function $\Sigma\, w(|F_o| - |F_c|)^2$, where $w = 1/\sigma_F{}^2$. Initially isotropic temperature factors were used, but in the final refinements all nonhydrogen atoms were varied assuming anisotropic thermal motion. The positions of 18 of the 21 hydrogen atoms could be found from ΔF maps. However, refinement of the hydrogen atom parameters led to chemically unreasonable bond lengths and angles. Hence the positions of all the hydrogen atoms were calculated (C-H = 0.95A) and their contributions included in the structure factor calculations, assuming an isotropic temperature factor, B, of 4.0A^2. The hydrogen atom parameters were not refined. The refinement converged with $R = \Sigma||Fo|-|Fc||/\Sigma|Fo| = 0.031$ and $R_w = (\Sigma w(|F_o|-|F_c|)^2/\Sigma w|Fo|^2)^{\frac{1}{2}} = 0.035$.

A correction for anomalous dispersion was made for all nonhydrogen atoms. Scattering factors were from Ref. 12. The osmium atom was assumed to be in the zero ionization state. No evidence of secondary extinction was found.

Attempts to apply absorption corrections were made. Transmission coefficients, calculated using a Gaussian integration method (6x4x6 grid), varied from 0.43 to 0.68 with most being about 0.6. The R factors increased substantially ($R=0.049$, $R_w=0.056$) when refinements were attempted using the corrected data. No improvement was noted in the standard deviations. It was concluded that the errors introduced in applying the corrections to a relatively small crystal enclosed in a capillary were larger than those introduced by ignoring absorption effects. Thus, the final refinements were carried out on uncorrected data.

In the last cycle of refinement all shifts on positional and thermal parameters were less than one standard deviation, with the largest shift being 0.37 standard deviations. The final value of the standard deviation of an observation of unit weight, defined as $[\Sigma w(|Fo|-|Fc|)^2/(N_o-N_v)]^{\frac{1}{2}}$ was 0.944 for N_o = 2820 reflections and N_v = 442 parameters.

There were two peaks of about 1.5 e/A^3 in the final difference Fourier. These were quite close to the osmium atom. Neither they nor any of the other peaks were considered physically significant.

Most calculations were performed on a PDP 11/40 computer using the Enraf-Nonius structure determination package (SDP). Johnson's ORTEP,[13] some molecular geometry calculations (using XANADU by Roberts and Sheldrick) and local programs were run on an Amdahl 470v/6 computer. Use was made of the PDP 11/40-Vector General graphics system.[14] Data reduction was performed on the Honeywell computer at the University of Houston.

The final positional and thermal parameters are given in Table II. The final calculated positions of the hydrogen atoms are given in Table III. Tables IV and V contain the root-mean-square components of thermal displacement along the principal axis of the thermal ellipsoids and the observed and calculated structure factors respectively. Tables III-V are available as supplementary material.

Results and Discussion

The following reactions for the synthesis of phthalocyanatoruthenium complexes have been carefully examined, and considerable discrepancies from the reported results[4] have been found. Furthermore, a new carbonyl complex of ruthenium phthalocyanine has been isolated from the reaction products. The reaction of $RuCl_3 \cdot 3(H_2O)$ with phthalonitrile was reported to give PcRu(III)Cl as the major product of the reaction by Berizin and Sennikova,[4,5] as shown in the following equation:

$$RuCl_3(H_2O) + C_6H_4(CN)_2 \rightarrow PcRu(III)Cl$$

The reaction of $RuCl_3 \cdot 3(H_2O)$ with o-cyanobenzoamide was reported to give PcRu(II) as its major product by Krueger and Kenny,[6a] as shown in the following equation:

$$RuCl_3 \cdot 3(H_2O) + NCC_6H_4CONH_2 \rightarrow PcRu(II)$$

From our investigations both of these reactions have been found to give PcRu(II) as the major product and PcRu(III)Cl as the minor one.

In addition, a small quantity of PcRu(II)(CO)L (L is a solvent used for the isolation) was detected in the crude $PcRu(II) \cdot 6C_6H_5NH_2$ according to its infrared spectrum, and was isolated by column chromatography. The yield of PcRu(II)(CO)L was less than 5%. Thus, the reactions above should be expressed by the following equations:

$$RuCl_3 \cdot 3(H_2O) + C_6H_4(CN)_2 \rightarrow PcRu(II) + PcRu(III)Cl + PcRu(II)(CO)L$$

$$RuCl_3 \cdot 3(H_2O) + NCC_6H_4CONH_2 \rightarrow PcRu(II) + PcRu(III)Cl + PcRu(II)(CO)L$$

The yield of 5% for CO complex is rather surprising in two respects. In the first place, the source of carbon monoxide is unknown. Since the reaction of $RuCl_3 \cdot 3(H_3O)$ with phthalonitrile was carried out in air without solvent there should not be any direct carbonyl sources in the system. In the second place, the major product was four-coordinate PcRu(II), whereas it appears that stable Ru(II) porphyrin complexes are generally six coordinate.[15-20]

This carbonyl complex of ruthenium phthalocyanine can be synthesized in tow different ways directly in high yield (80-90%). The reaction of $RuCl_3 \cdot 3(H_2O)$ with excess phthalonitrile under carbon monoxide atmosphere gives an almost quantitative yield even after the isolation of the compound through column chromatography. The reaction of $Ru_3(CO)_{12}$ with excess phthalonitrile also gives a high yield of PcRu(II)(CO)(Py). This reactions are shown in the following equations:

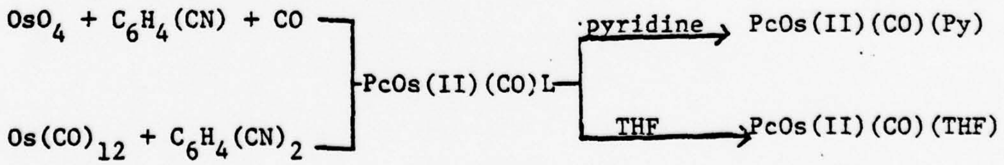
$$RuCl_3 \cdot 3(H_2O) + C_6H_4(CN)_2 + CO \rightarrow PCRu(II)(CO)L \xrightarrow{pyridine} PcRu(II)(CO)(Py)$$

$$Ru_3(CO)_{12} + C_6H_4(CN)_2 \longrightarrow RcRu(II)(CO)L \xrightarrow{pyridine} PcRu(II)(CO)(Py)$$

The carbonyl complex of ruthenium phthalocyanine was characterized by infrared and visible absorption spectroscopy and elemental analysis. The infrared spectrum of the compound shows an intense band at 1965 cm^{-1} which is assigned to $\nu C \equiv O$ attached to ruthenium ion.

A well-defined carbonyl complex of osmium phthalocyanine was also isolated from the products of the reported reactions of OsO_4 with molten phthalonitrile.[5] Although the yield of PcOs(II)(CO)L is less than 5%, it it isolated as a pure complex by column chromatography. The isolation was made possible by the remarkable solubility of the complex in common organic solvents. The presence of a carbonyl ligand attached to osmium metal was evidenced by an intense band at 1930 cm^{-1} in the infrared spectrum. This new type of compound, PcOs(II)(CO)L, coordinates another donor molecule as its last axial ligand. For example, tetrahydrofuran and pyridine can coordinate to the open sixth site of osmium to form a stable compound with octahedral configuration.

These carbonyl complexes of osmium phthalocyanine can be prepared in high yield (80-90%) by the reaction of OsO_4 with molten phthalonitrile in carbon monoxide atmosphere or the reaction of $Os_3(CO)_{12}$ with phthalonitrile in air. Subsequent treatment with pyridine or THF yield PcOs(II)(CO)(Py) and PcOs(II)(CO)(THF) respectively. Column chromatography gives a single blue band of each complex. An x-ray diffraction analysis has been carried out on the former complex.



Carbonyl complexes of ruthenium and osmium porphines have been synthesized and characterized by a number of workers.[15-20]

Table VI summarized the carbonyl stretching frequencies of these complexes together with those of the carbonyl complexes of ruthenium and osmium phthalocyanocines.

(Tetraphenylporphinato)(carbonyl)(pyridine)ruthenium(II) (TPPRu(CO)(Py)) shows a carbonyl peak at 1939 cm^{-1}, which is slightly lower than that (1965 cm^{-1}P of PcRu(CO)(Py). Therefore, the back donation of 4d electrons of ruthenium to the anti-bonding π-orbitals of the carbonyl ligand seems to be less in the phthalocyanine complex than in the TPP complex. The same phenomenon is observed in osmium complexes. (Octaethylporphinato)(Carbonyl)(pyridine)Os(II) (OEPOs(CO)(Py)) shows a carbonyl peak at 1902 cm^{-1}, while PcOs(CO)(Py) shows a carbonyl peak at 1930^{-1}. In this case back donation of 5d electrons from osmium to the anti-bonding π-orbital of the carbonyl ligand seems to be less in the phthalocyanine than in the OEP complex. Probably the structural difference of the rings causes the difference in the degree of the back donoation of d-electrons from the central metal ions to the coordinated carbon monoxide. Apparently, Ru(II) or Os(II) in the phthalocyanine carbonyl complexes donate less electron density to the carbonyl ligand than those in porphyrin rings.

It should also be noted that the PcRu(II) complex, which does not have a carbonyl ligand, is capable of coordinating carbon monoxide at room temperature under 1 atmosphere of carbon monoxide. PcRu(II) can be prepared by removing aniline molecules from $PcRu(II)\cdot 6(C_6H_5NH_2)$ under vacuum at high temperature (200°C) and it can form a carbonyl complex in THF or pyridine by bubbling CO gas into the solution for one day. The original PcRu(II)·6($C_6H_5NH_2$) can be recovered by refluxing PcRu(II) in freshly distilled aniline for a few minutes as shown in the following equations:

$$\begin{tikzcd}
 & & \mathrm{PcRu(II)(CO)(Py)} \\
\mathrm{PcRu(II)\cdot 6(C_6H_5NH_2)} \arrow[r, shift left, "\Delta\mathrm{,\ vacuum}"] & \mathrm{PcRu(II)} \arrow[l, shift left, "\Delta\mathrm{,\ C_6H_5NH_2}"] \arrow[ur, "\mathrm{CO\ in\ pyridine}"] \arrow[dr, "\mathrm{CO\ in\ THF}"'] & \\
 & & \mathrm{PcRu(II)(CO)(THF)}
\end{tikzcd}$$

The carbonylation process can be traced by noting changes in its visible spectrum as shown in Fig. I.

PcRu(II) is expected to demonstrate an interesting chemistry, because it does not possess a carbonyl ligand. On the other hand, the chemistry of ruthenium porphyrin is rather limited becasue removal of the carbonyl ligand is extremely difficult.[16,21] PcRu(II) appears to have the potential to form new ruthenium phthalocyanine complexes with various other molecules such as O_2, N_2, NO, and olefins. Also, the remarkable solubility of ruthenium and osmium phthalocyanine complexes will possibly open a new era in the coordination chemistry of metallophthalocyanines.

DESCRIPTION OF STRUCTURE OF PcOs(CO)(Py)

The structure of (carbonyl)(pyridine)phthalocyanatoosmium(II) is shown in Figure 2. Stereoviews are shown in Figures 3 and 4. The osmium ion is octahederally coordinated with the carbonyl and pyridine groups axially coordinated. The pyridine ring is tilted slightly with respect to the perpendicular to the phthalocyanine ring. The interplanar angle is 98.6°.

The structure of an analogous osmium porphyrin complex has not been reported, but the structure of (carbonyl)(pyridine)tetraphenylporphinatoruthenium(II), TPPRu(CO)(Py) has been published.[22] Osmium and ruthenium have similar covalent radii.[23] The phthalocyanine complex bears a strong resemblance to this metalloporphine. In addition, the structure of a carbonyl osmium porphodimethene complex with pyridine as the other axial ligand, $OEPMe_2Os(CO)(Py)$ has been reported.[24] Interesting comparisons may be made between these two complexes and PcOs(CO)(Py).

Bond lengths and angles are given in Table VII. The average interatomic distances between the osmium atom and the isoindole nitrogen atoms of the phthalocyanine molecule is 2.01A. However, it should be noted that the bond lengths for Os-N(2) and Os-N(6) are 1.98A, while the other two distances are 2.03A. The difference corresponds to ~5 standard deviations, so it is statistically significant. The C-N-C and N-C-C angles involving the isoindole nitrogen atoms also differ in the two pairs of isoindole groups. Thus, for groups 1 and 3 (containing N(2) and N(6), the average C-N-C angle is 112°, while the N-C-C an is 107°. In groups 2 and 4 (containing N(4) and N(8)) the corresponding angles are 107° and 111°. The differences once again correspond to about 4 or 5 standard deviations and are therefore significant. We are unable to offer a plausible explanation for these differences, which are not observed in other phthalocyanine complexes.

The size of the central "hole" in phthalocyanine complexes has been estimated to be 0.046 -0.050A smaller than in corresponding porphyrin complexes.[25] A similar difference is noted in the present case. The osmium atom is slightly out of the plane of the phthalocyanine molecule, so the size of the hole has to be estimated from the distances between opposite isoindole nitrogen atoms. These distances are 4.06 and 3.96A, corresponding to an average radius (Ct-N distance) of 2.01A for the macrocyclic hole. In TPPRu(CO)(Py)[22] the corresponding distance is 2.05A. The M-N distances are correspondingly longer. In both TPPRu(CO)(Py)[22] and $OEPMe_2Os(CO)(Py)$[24] these average 2.06A.

The Os-N bond lengths involving the isoindole nitrogen atoms are short for such bonds. On the other hand the $Os\text{-}N_{Py}$ (N_{Py} is the pyridine nitrogen atom) distance of 2.209(9)A is relatively long. $Ru\text{-}N_{Py}$ distances of 2.06-2.09Å have been reported.[26] $M\text{-}NH_3$ bond lengths in amine complexes of Os(II) and Ru(II) range from 2.12-2.14A.[26-29] The $M\text{-}N_{Py}$ distance in both TPPRu(CO)(Py) and $OEPMe_2Os(CO)(Py)$ is also elongated (2.193(4) and 2.230(4)A respectively.)

This long bond may be due to steric interactions between atoms in the pyridine ring, particularly the α-hydrogen atoms, and atoms in the macrocycle. This would block a closer approach to the osmium atom by the pyridine molecule. The osmium atom lies 0.15A on the other side of phthalocyanine molecule. However, the closest contacts involving the α-hydrogen atoms are between H(33) and C(32) and between H(37) and C(16). These contacts are both about 2.73A, which is not an unusually short contact distance in such cases. A tabulation of such contacts in porphyrin complexes containing planar axial ligands shows a range of 2.45-2.9A.[30] In TPPRu(CO)(Py) the closest contact is 2.51A.

The angle between the plane of the pyridine ring and the plane defined by N(2), Os, and N(6) is 48°. An angle of ~45° minimizes steric interaction, whereas an angle of ~0° would bring the α-hydrogen atoms into close contact with the isoindole nitrogen atoms.[31]

It has also been suggested a lengthening of ~0.1A will occur in the axial M-N bond due to the trans effect of the carbonyl group.[22] In OEPRu(Py)$_2$ [32] the observed axial bond lenghts are indeed shorter, 2.09-2.10A, in spite of some relatively close contacts of the α-hydrogen atoms of the pyridine ligands with atoms in the macrocycle.[30] An even shorter M-N bond length of 2.00A has been reported in PcFe(Py)$_2$.[33]

The metal carbonyl distance of 1.83(1) is in good agreement with the values found in TPPRu(CO)(Py) OEPMe$_2$Os(CO)(Py). The Os-C-O angle is linear (177°).

Least squares plane of interest are given in Table VIII. As mentioned previously, the osmium atom is 0.15A out of the plane defined by the phthalocyanine group in a direction toward the carbonyl group. The displacement decreases to 0.099A if one considers only the plane of the four isoindole nitrogen atoms. By way of comparison, the ruthenium atom in TPPRu(CO)(Py) is 0.079A out of the plane of the porphyrin skeleton toward the carbonyl group.

In the discussion on TPPRu(CO)(Py) the authors attributed the out-of-plane displacement of the metal ion to either very strong metal-carbonyl bonds or to the inability of the large metal ion to fit into the plane of the macrocycle.[22] The former possibility now appears more likely, since in OEPRu(Py)$_2$ the ruthenium(II) ion lies in the plane of the macrocycle. The same factor is probably primarily responsible for the out-of-plane displacement observed for the metal ion in this phtalocyanine complex.

The difference of 0.065A in the displacements of the metal ion from the planes of the macrocycle and of the isoindole nitrogen atoms indicate that the macrocycle itself is "domed", corresponding to a C_{4v} deviation from planarity. Deviations of equal or larger magnitude have been observed in other metallophthalocyanine complexes, where the metal ion is out of the plane of the macrocycle.[34-37] The difference between the deviation of the osmium atom from the plane of the four isoindole nitrogen atoms and the plane defined by the pyrrole rings and the bridging nitrogen atoms is 0.027A, a value falling in the middle of the range found for porphyrins with the metal ion out of the macrocyclic plane.[38]

The doming is not equal for the four isoindole groups. The maximum deviations from the plane of the four isoindole nitrogen atoms are 0.23 and 0.35A for phenyl carbon atoms in groups 2 and 3, while the maximum deviations in the same direction in groups 1 and 4 are 0.04 and 0.12A. Such a pattern was observed in aquophthalocyanatomagensium(II).[36]

There appears to be some variation in bond parameters of phthalocyanine complexes as the size of the central "hole" increases.[39] The C-N-C angle involving the azamethine nitrogen atom is the most sensitive bond parameter. This angle ranges from 121.7° in Fe(Pc)[25] (Ct-N: 1.93A) to 126.2° in $Cl_2Sn(Pc)$[40] (Ct-N: 2.05A). The average value of 125(1)° found for this angle in PcOs(CO)(Py) agrees with that found in phthalocyanine complexes with similar Ct-N distances.[35,39] The presence of a very heavy metal atom like osmium decreases the accuracy with which the lighter atoms can be determined, but within the observed standard deviations, the other bond parameters agree with those reported in the accurately determined structure of Zn(Pc)[39] and Sn(Pc).[34]

A packing diagram of the unit cell is shown in Figure 3. Table IX lists the intermolecular contacts $\leq$ 3.5A. Most of the shortest contacts involve the carbonyl oxygen atom. Non-bonded contacts of this magnitude have been observed in other carbonyl complexes (e.g. $[H_3OEP]^+$ $[Re_2(CO)_6Cl_3]^-$).[41] Neither these nor any of the other intermolecular contacts are believed to have any significant effect on the structure.

The structure of PcRu(CO)(Py) probably possesses a structure similar to that of OsPc(CO)Py.

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Supplementary Material Available: Table III-V containing calculated hydrogen atom positions, root-mean-square components of thermal ellipsoids and observed and calculated structure factors (pages). Ordering information is given on any current masthead page.

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TABLE I

Crystal Data for $(C_{38}H_{21}N_8)Os$[a]

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a = 11.966(9) A

b = 15.705(3) A

c = 17.749(5) A

B = 107.92 (4)°

V = 3125(4) A^3

Fw = 809.9

Z = 4

d calcd = 1.721

μ = 43.9 cm^{-1} (MoK α radiation)

Systematic absences h0l (h+l odd)
0k0 (k odd)

Space group $P\,2_1/n$

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[a] In this and subsequent tables the estimated standard deviation of the least significant figures shown in parentheses.

Table II

Table II Fractional Coordinates and Thermal Motion Parameters Derived from the Least-Squares Refinement

ATOM	X	Y	Z	U(11)	U(22)	U(33)	U(12)	U(13)	U(23)
OS	0.22473(4)	0.11171(3)	0.11752(3)	352(2)	303(2)	276(2)	-1(7)	274(3)	61(6)
N(1)	0.4585(7)	0.1150(6)	0.0454(4)	33(4)	41(5)	37(4)	0(12)	31(7)	1(11)
N(2)	0.2651(7)	0.1753(6)	0.0286(4)	31(4)	34(5)	31(4)	1(11)	27(7)	31(11)
N(3)	0.0855(7)	0.2557(5)	-0.0241(5)	42(5)	34(6)	26(5)	-5(10)	16(8)	17(9)
N(4)	0.0711(7)	0.1710(6)	0.0896(4)	32(5)	47(6)	23(4)	-5(10)	21(7)	33(9)
N(5)	-0.0051(6)	0.1119(6)	0.1914(4)	33(4)	32(5)	28(4)	8(11)	22(7)	-9(10)
N(6)	0.1925(7)	0.0575(5)	0.2143(4)	38(5)	29(5)	28(4)	-6(9)	33(7)	15(8)
N(7)	0.3804(7)	-0.0112(5)	0.2739(5)	33(5)	28(5)	43(5)	13(9)	33(8)	16(9)
N(8)	0.3844(7)	0.0620(6)	0.1521(4)	37(5)	47(6)	24(4)	-10(10)	25(8)	32(9)
C(1)	0.3667(9)	0.1631(7)	0.0084(6)	48(7)	38(7)	30(6)	-31(13)	35(10)	-21(11)
C(2)	0.3569(10)	0.2151(7)	-0.0625(6)	55(7)	27(6)	26(5)	-27(12)	37(10)	-8(10)
C(3)	0.4347(10)	0.2275(8)	-0.1073(6)	52(7)	48(8)	46(6)	-14(13)	58(10)	-4(13)
C(4)	0.3991(11)	0.2840(8)	-0.1704(6)	81(8)	56(8)	45(6)	-20(15)	86(10)	11(13)
C(5)	0.2923(11)	0.3273(8)	-0.1889(6)	96(9)	47(8)	39(6)	-50(15)	51(12)	21(13)
C(6)	0.2155(10)	0.3135(7)	-0.1460(6)	62(8)	39(7)	33(6)	-14(13)	40(11)	4(12)
C(7)	0.2483(9)	0.2568(7)	-0.0826(5)	50(7)	34(7)	22(5)	-13(12)	29(10)	1(11)
C(8)	0.1908(9)	0.2290(7)	-0.0237(6)	50(7)	30(7)	29(6)	-14(12)	36(10)	5(11)
C(9)	0.0300(9)	0.2300(7)	0.0282(6)	40(7)	30(7)	34(6)	-4(12)	18(10)	-5(11)
C(10)	-0.0855(10)	0.2601(7)	0.0268(6)	51(7)	34(7)	27(6)	10(13)	20(10)	-1(11)
C(11)	-0.1664(10)	0.3177(7)	-0.0210(6)	62(8)	41(7)	39(7)	33(14)	18(12)	24(12)
C(12)	-0.2715(10)	0.3290(8)	-0.0091(7)	46(7)	56(8)	65(8)	61(14)	20(13)	17(15)
C(13)	-0.3023(10)	0.2830(8)	0.0492(7)	51(7)	52(8)	66(8)	44(14)	40(12)	7(14)
C(14)	-0.2244(9)	0.2267(7)	0.0985(6)	34(6)	49(8)	35(6)	16(12)	25(10)	-2(12)
C(15)	-0.1160(10)	0.2150(7)	0.0874(6)	46(7)	25(6)	36(6)	10(12)	19(11)	-9(11)
C(16)	-0.0135(9)	0.1622(7)	0.1269(5)	38(6)	23(6)	23(5)	15(11)	6(10)	15(10)
C(17)	0.0883(9)	0.0669(7)	0.2311(6)	28(6)	36(7)	29(5)	-9(11)	29(9)	-5(11)
C(18)	0.1005(9)	0.0206(6)	0.3057(6)	37(6)	22(6)	28(6)	1(11)	22(9)	12(10)
C(19)	0.0226(9)	0.0057(7)	0.3493(6)	47(6)	38(7)	39(6)	-2(12)	55(9)	-5(11)
C(20)	0.0643(10)	-0.0424(7)	0.4189(6)	76(7)	39(7)	45(6)	0(13)	90(10)	15(12)
C(21)	0.1783(11)	-0.0752(7)	0.4443(6)	77(8)	46(8)	46(6)	9(14)	70(11)	29(12)
C(22)	0.2542(9)	-0.0603(7)	0.4014(6)	40(7)	50(8)	36(7)	13(13)	22(11)	4(12)
C(23)	0.2153(10)	-0.0139(7)	0.3308(5)	58(7)	26(6)	23(5)	5(12)	32(10)	14(10)
C(24)	0.2726(10)	0.0111(6)	0.2707(6)	52(7)	26(6)	24(6)	13(12)	21(10)	15(10)
C(25)	0.4314(9)	0.0105(7)	0.2180(6)	33(6)	32(7)	41(6)	-12(12)	28(10)	2(12)
C(26)	0.5508(9)	-0.0171(7)	0.2209(6)	34(6)	29(7)	40(6)	8(12)	19(10)	2(11)
C(27)	0.6338(9)	-0.0660(8)	0.2741(6)	34(6)	55(8)	50(7)	18(13)	35(10)	13(13)
C(28)	0.7382(10)	-0.0813(7)	0.2582(7)	38(7)	40(7)	72(8)	23(12)	32(12)	12(13)
C(29)	0.7596(10)	-0.0452(7)	0.1915(7)	38(7)	41(8)	65(7)	17(13)	43(11)	-12(13)
C(30)	0.6756(10)	0.0044(7)	0.1377(6)	48(7)	33(7)	52(7)	0(12)	46(10)	1(12)
C(31)	0.5704(9)	0.0193(7)	0.1533(6)	29(6)	33(7)	44(6)	-6(11)	29(10)	-2(11)
C(32)	0.4680(9)	0.0061(7)	0.1120(6)	43(7)	31(6)	29(6)	11(12)	14(10)	19(11)
N(9)	0.3026(7)	0.2178(5)	0.1988(5)	37(5)	37(5)	27(5)	-15(10)	23(8)	-1(9)
C(33)	0.4083(9)	0.2481(7)	0.2054(6)	31(6)	37(7)	40(6)	-15(12)	14(10)	-14(12)
C(34)	0.4642(11)	0.3067(8)	0.2613(7)	52(8)	46(8)	65(8)	-6(14)	14(14)	3(14)
C(35)	0.4107(11)	0.3387(7)	0.3133(7)	61(8)	32(7)	48(7)	-14(14)	0(13)	-14(13)
C(36)	0.2990(11)	0.3112(7)	0.3062(6)	84(9)	36(7)	41(6)	-13(14)	58(12)	-24(12)
C(37)	0.2474(10)	0.2520(7)	0.2489(6)	44(6)	32(6)	55(7)	-19(13)	49(10)	8(12)
C(38)	0.1629(9)	0.0225(8)	0.0509(6)	50(6)	97(9)	27(5)	-80(14)	66(9)	-33(13)
O(1)	0.1273(8)	-0.0374(5)	0.0109(5)	95(7)	64(6)	67(5)	-35(11)	56(10)	-59(10)

Table II cont'd.

[a] The Debye-Waller Factor is defined as $T=\exp[-2\pi^2(U_{11}a^{*2}h^2+U_{22}b^{*2}k^2+U_{33}c^{*2}l^2+U_{12}a^*b^*hk+U_{13}a^*c^*hl+U_{33}b^*c^*kl)]$. The values for U have been multiplied by 10^3, except for those of Os, which have been multiplied by 10^4.

Table VI. Comparison of CO Frequencies of MP(CO)(Py)

PcM(CO)(Py)	RuPc	RuTPP	OsPc	OsTPP	OsOEP
$\nu C\equiv O(cm^{-1})$	1965	1939	1930	1920	1902

P = Pc, TPP, and OEP

Pc = phthalocyanine

TPP = tetraphenylporphine

OEP = octaethylporphyrin

Py = pyridine

M = Os or Ru

TABLE VII

Bond Lengths (Å) and Angles (deg)^a

Os-N(2)	2.027(9)	2.01(3)	N(2)-Os-N(4)	90.1(4)	89.9(3)
Os-N(4)	1.983(9)		N(2)-Os-N(8)	89.6(4)	
Os-N(6)	2.034(9)		N(4)-Os-N(6)	90.1(4)	
Os-N(8)	1.978(9)		N(6)-Os-N(8)	89.7(4)	
Os-N(9)	2.202(9)		N(2)-Os-N(6)	174.2(4)	174.3(2)
Os-C(38)	1.83(1)		N(4)-Os-N(8)	174.5(4)	
N(2)-C(1)	1.38(1)	1.37(1)	N(2)-Os-N(9)	88.2(4)	87(1)
N(2)-C(8)	1.36(1)		N(4)-Os-N(9)	88.8(4)	
N(4)-C(9)	1.39(1)		N(6)-Os-N(9)	86.0(4)	
N(4)-C(16)	1.37(1)		N(8)-Os-N(9)	85.7(4)	
N(6)-C(17)	1.37(1)		N(2)-Os-C(38)	91.9(5)	93(1)
N(6)-C(24)	1.36(1)		N(4)-Os-C(38)	92.3(5)	
N(8)-C(25)	1.38(1)		N(6)-Os-C(38)	93.8(5)	
N(8)-C(32)	1.39(1)		N(8)-Os-C(38)	93.2(5)	
N(1)-C(1)	1.33(1)	1.34(2)	N(9)-Os-C(38)	178.9(6)	
N(1)-C(32)	1.33(1)		Os-N(2)-C(1)	124.3(8)	125(2)
N(3)-C(8)	1.35(1)		Os-N(2)-C(8)	124.3(8)	
N(3)-C(9)	1.36(1)		Os-N(4)-C(9)	125.8(8)	
N(5)-C(16)	1.32(1)		Os-N(4)-C(16)	126.9(7)	
N(5)-C(17)	1.32(1)		Os-N(6)-C(17)	123.3(8)	
N(7)-C(24)	1.35(1)		Os-N(6)-C(24)	123.8(9)	
N(5)-C(25)	1.37(1)		Os-N(8)-C(25)	126.4(8)	
C(1)-C(2)	1.46(1)	1.46(2)	Os-N(8)-C(32)	127.3(8)	
C(7)-C(8)	1.47(1)		C(1)-N(2)-C(8)	111(1)	109(3)
C(9)-C(10)	1.45(2)		C(9)-N(4)-C(16)	107(1)	
C(15)-C(16)	1.47(1)		C(17)-N(6)-C(24)	113(1)	
C(17)-C(18)	1.46(1)		C(25)-N(8)-C(32)	106(1)	
C(23)-C(24)	1.47(2)				
C(25)-C(26)	1.48(2)		C(1)-N(1)-C(32)	125(1)	125(1)
C(31)-C(32)	1.42(2)		C(8)-N(3)-C(9)	126(1)	
C(2)-C(7)	1.40(2)	1.41(1)	C(16)-N(5)-C(17)	124(1)	
C(10)-C(15)	1.42(1)		C(24)-N(7)-C(25)	125(1)	
C(18)-C(23)	1.41(2)		N(1)-C(1)-N(2)	128(1)	127(2)
C(26)-C(31)	1.40(2)		N(2)-C(8)-N(3)	125(1)	
C(2)-C(3)	1.40(2)	1.39(1)	N(3)-C(9)-N(4)	126(1)	
C(6)-C(7)	1.38(1)		N(4)-C(16)-N(5)	125(1)	
C(10)-C(11)	1.40(2)		N(5)-C(17)-N(6)	128(1)	
C(14)-C(15)	1.38(2)		N(6)-C(24)-N(7)	129(1)	
C(18)-C(19)	1.39(1)		N(7)-C(25)-N(8)	127(1)	
C(22)-C(23)	1.39(1)		N(8)-C(32)-N(1)	125(1)	
C(26)-C(27)	1.37(2)		N(1)-C(1)-C(2)	125(1)	124(1)
C(30)-C(31)	1.39(2)		N(3)-C(8)-C(7)	123(1)	
C(3)-C(4)	1.38(2)	1.38(1)	N(3)-C(9)-C(10)	124(1)	
C(5)-C(6)	1.37(2)		N(5)-C(16)-C(15)	124(1)	
C(1)-C(12)	1.35(2)		N(5)-C(17)-C(18)	125(1)	
C(13)-C(14)	1.38(2)		N(7)-C(24)-C(23)	124(1)	
C(19)-C(20)	1.39(2)		N(7)-C(25)-C(26)	123(1)	
C(21)-C(22)	1.36(2)		N(1)-C(32)-C(31)	124(1)	
C(27)-C(128)	1.38(2)				
C(29)-C(130)	1.39(2)				

Table VII cont'd.

C(4)-C(5)	1.40(2)	1.39(1)
C(12)-C(13)	1.39(2)	
C(20)-C(21)	1.40(2)	
C(28)-C(29)	1.39(2)	
N(9)-C(33)	1.32(1)	1.34(3)
N(9)-C(37)	1.36(2)	
C(33)-C(34)	1.36(2)	1.36(1)
C(34)-C(35)	1.36(2)	
C(35)-C(36)	1.37(2)	
C(36)-C(37)	1.37(2)	
C(38)-O(1)	1.17(1)	
N(2)-N(4)	2.84(1)	2.83(1)
N(2)-N(8)	2.82(1)	
N(4)-N(6)	2.84(1)	
N(6)-N(8)	2.83(1)	
N(2)-N(6)	4.06(1)	4.01(7)
N(4)-N(8)	3.96(1)	
N(1)-N(5)	6.78(1)	6.78(1)
N(3)-N(7)	6.77(1)	

N(2)-C(1)-C(2)	107(1)	109(2)
N(2)-C(8)-C(7)	108(1)	
N(4)-C(9)-C(10)	110(1)	
N(4)-C(16)-C(15)	111(1)	
N(6)-C(17)-C(18)	106(1)	
N(6)-C(24)-C(23)	106(1)	
N(8)-C(25)-C(26)	110(1)	
N(8)-C(32)-C(31)	111(1)	
C(1)-C(2)-C(7)	108(1)	107(1)
C(2)-C(7)-C(8)	106(1)	
C(9)-C(10)-C(15)	107(1)	
C(10)-C(15)-C(16)	105(1)	
C(17)-C(18)-C(23)	107(1)	
C(18)-C(23)-C(24)	107(1)	
C(25)-C(26)-C(31)	105(1)	
C(26)-C(31)-C(32)	107(1)	
C(1)-C(2)-C(3)	131(1)	132(1)
C(6)-C(7)-C(8)	132(1)	
C(9)-C(10)-C(11)	134(1)	
C(14)-C(15)-C(16)	134(1)	
C(17)-C(18)-C(19)	132(1)	
C(22)-C(23)-C(24)	132(1)	
C(25)-C(26)-C(27)	132(1)	
C(30)-C(31)-C(32)	133(1)	
C(7)-C(2)-C(3)	121(1)	121(1)
C(2)-C(7)-C(6)	121(1)	
C(15)-C(10)-C(11)	119(1)	
C(10)-C(15)-C(14)	121(1)	
C(23)-C(18)-C(19)	121(1)	
C(18)-C(23)-C(22)	120(1)	
C(31)-C(26)-C(27)	122(1)	
C(26)-C(31)-C(30)	120(1)	
C(2)-C(3)-C(4)	116(1)	118(1)
C(5)-C(6)-C(7)	118(1)	
C(10)-C(11)-C(12)	120(1)	
C(13)-C(14)-C(15)	119(1)	
C(18)-C(19)-C(20)	117(1)	
C(21)-C(22)-C(23)	119(1)	
C(26)-C(27)-C(28)	118(1)	
C(29)-C(30)-C(31)	118(1)	
C(3)-C(4)-C(5)	122(1)	121(1)
C(4)-C(5)-C(6)	121(1)	
C(11)-C(12)-C(13)	121(1)	
C(12)-C(13)-C(14)	121(1)	
C(19)-C(20)-C(21)	122(1)	
C(20)-C(21)-C(22)	121(1)	
C(27)-C(28)-C(29)	121(1)	
C(28)-C(29)-C(30)	121(1)	
Os-N(9)-C(33)	122.2(9)	121.8(5)
Os-N(9)-C(37)	121.5(8)	
C(33)-N(9)-C(38)	116(1)	
N(9)-C(33)-C(34)	124(1)	123(1)
N(9)-C(37)-C(36)	123(1)	
C(33)-C(34)-C(35)	120(1)	119(1)
C(34)-C(35)-C(36)	118(1)	
C(35)-C(36)-C(37)	119(1)	
Os-C(38)-O(1)	177(1)	

[a] Some nonbonded distances of interest are also given. Figures in parenthesis for the averaged values are the root-mean-square standard deviations of the least significant figure

TABLE VIII

Least-Squares Planes

A. Deviations (a) from planes

	Plane 1	Plane 2	Plane 3	Plane 4	Plane 5	Plane 6	Plane 7
Os	-0.154	-0.099	-0.083	-0.056	0.017	-0.070	
N(1)	-0.082	-0.102	0.044	-0.147	-0.001	0.088	
N(2)	-0.006	0.003	0.025	-0.086	0.248	0.181	
N(3)	0.053	0.074	-0.026	-0.111	0.511	0.297	
N(4)	-0.075	-0.003	-0.090	-0.018	0.251	0.039	
N(5)	-0.157	-0.029	-0.143	0.102	0.102	-0.158	
N(6)	-0.003	0.014	0.180	-0.013	-0.013	-0.117	
N(7)	0.067	0.156	0.288	0.424	-0.053	-0.009	
N(8)	-0.041	-0.004	0.113	0.096	-0.028	0.011	
C(1)	-0.049	-0.068	0.014	-0.176	0.148	0.160	
C(2)	-0.029	-0.075	-0.011	-0.287	0.261	0.258	
C(3)	-0.042	-0.121	-0.016	-0.390	0.240	0.295	
C(4)	0.030	-0.069	0.000	-0.437	0.418	0.437	
C(5)	0.117	0.030	0.028	-0.379	0.615	0.546	
C(6)	0.097	0.043	0.001	-0.309	0.603	0.478	
C(7)	0.020	-0.013	-0.022	-0.267	0.420	0.329	
C(8)	0.014	0.016	-0.018	-0.157	0.389	0.258	
C(9)	0.025	0.078	-0.047	-0.034	0.462	0.209	
C(10)	0.070	0.144	-0.054	0.018	0.600	0.255	
C(11)	0.159	0.228	-0.031	0.021	0.813	0.397	
C(12)	0.111	0.205	-0.115	0.009	0.830	0.333	
C(13)	-0.051	0.075	-0.250	-0.031	0.612	0.102	
C(14)	-0.118	0.013	-0.253	-0.011	0.422	-0.021	
C(15)	-0.062	0.044	-0.159	0.010	0.412	0.052	
C(16)	-0.102	0.001	-0.130	0.035	0.244	-0.031	
C(17)	-0.124	0.002	-0.048	0.196	0.018	-0.166	
C(18)	-0.037	0.118	0.086	0.416	0.014	-0.157	
C(19)	-0.066	0.122	0.049	0.478	-0.009	-0.236	
C(20)	0.039	0.246	0.208	0.699	-0.012	-0.203	
C(21)	0.157	0.353	0.386	0.845	-0.004	-0.106	
C(22)	0.187	0.351	0.423	0.787	0.021	-0.026	
C(23)	0.062	0.205	0.244	0.542	0.001	-0.080	
C(24)	0.021	0.128	0.195	0.387	-0.016	-0.055	
C(25)	0.005	0.060	0.216	0.256	-0.093	-0.012	
C(26)	0.014	0.049	0.279	0.259	-0.178	-0.003	
C(27)	0.083	0.122	0.411	0.412	-0.232	0.011	
C(28)	0.046	0.059	0.411	0.337	-0.333	-0.008	
C(29)	-0.010	-0.027	0.328	0.159	-0.329	0.008	
C(30)	-0.079	-0.102	0.194	0.003	-0.275	-0.007	
C(31)	-0.052	-0.049	0.184	0.069	-0.183	0.003	
C(32)	-0.099	-0.094	0.070	-0.041	-0.106	-0.003	
N(9)							0.020
C(33)							-0.013
C(34)							-0.004
C(35)							0.013
C(36)							-0.005
C(37)							-0.011

B. Angles (deg) between Least-Squares Planes

	Plane 2	Plane 3	Plane 4	Plane 5	Plane 6	Plane 7
Plane 1	1.4	2.7	5.2	5.1	3.1	98.6
Plane 2		3.0	4.1	5.5	4.2	98.0
Plane 3			4.1	7.9	5.3	96.1
Plane 4				9.4	8.3	94.1
Plane 5					3.7	103.4
Plane 6						101.4

C. Equations of Planes[a]

Plane 1 Marcocycle; N(1)N(8), C(1)-C(32)
$2.721x + 12.314y + 8.382z = 3.126$

Plane 2 Isoindole nitrogen atoms; N(2), N(4), N(6), N(8)
$2.509x + 12.216y + 8.726z = 3.053$

Plane 3 Isoindole Group I; N(2), C(1)-C(8)
$3.051x + 11.869y + 8.660z = 3.113$

Plane 4 Isoindole Group 2; N(4), C(9)-C(16)
$2.438x + 11.516y + 9.693z = 3.029$

Plane 5 Isoindole Group 3; N(6), C(17)-C(24)
$2.125x + 13.099y + 7.731z = 2.833$

Plane 6 Isoindole Group 4; N(8), C(25)-C(32)
$2.836x + 12.756y + 7.589z = 3.024$

Plane 7 Pyridine Ring; N(9), C(33)-C(37)
$2.361x - 11.469y + 9.817z = 0.148$

---

[a] All planes are unweighted. x,y,z are in monoclinic fractional coordinates.

TABLE IX

Intermolecular contacts (A) ≤3.5A[a]

C(16)-O(1)^{I}	3.08	N(3)-C(35)^{II}	3.32
O(1)-O(1)^{I}	3.18	N(5)-C(4)^{III}	3.40
N(4)-O(1)^{I}	3.25	C(23)-C(35)^{IV}	3.41
C(15)-O(1)^{I}	3.26	C(24)-C(36)^{IV}	3.42
C(38)-O(1)^{I}	3.31		

[a] Roman numeral superscripts denote the following equivalent positions relative to the reference molecule at x,y,z:

I	$-x, -y, -z$
II	$-\frac{1}{2}+x, \frac{1}{2}-y, -\frac{1}{2}+z$
III	$-\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}-z$
IV	$1-x, -y, -z$

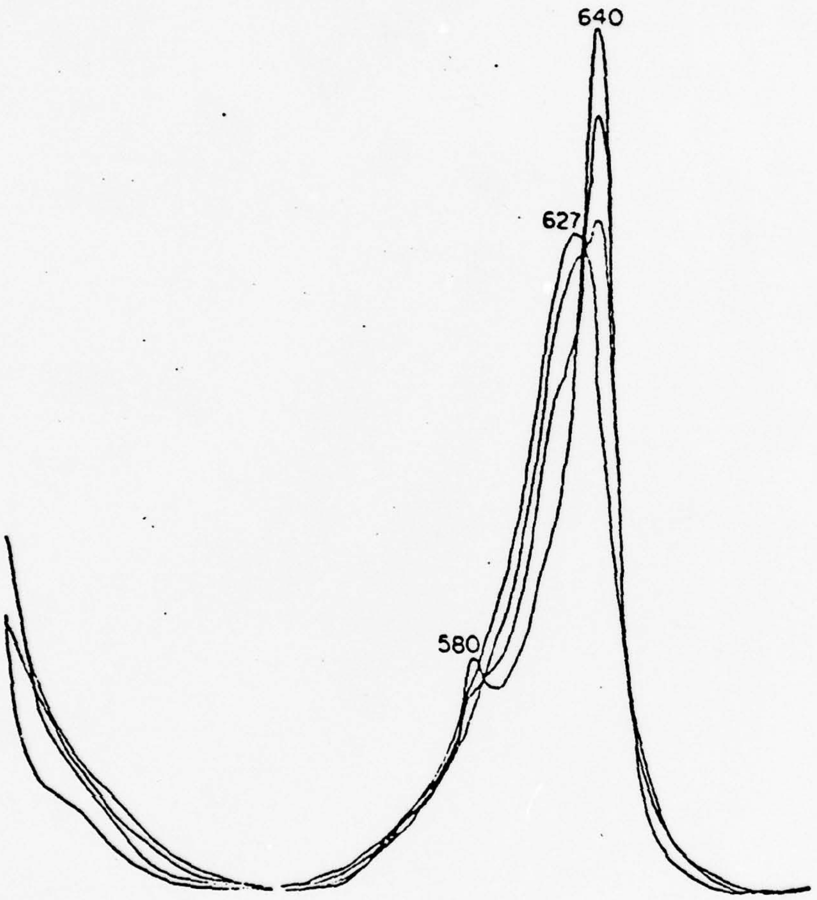

Fig. 1 Tsutsui

Figure 1. Coordination of carbon monoxide by PcRu(II) in THF. The peak at 627 nm is characteristic for PcRu(II) and the peaks at 640 and 580 nm are due to PcRu(CO)(THF). Upon coordination of carbon monoxide, the peak at 627 disappears, while new peaks (at 540 and 580 nm) appear.

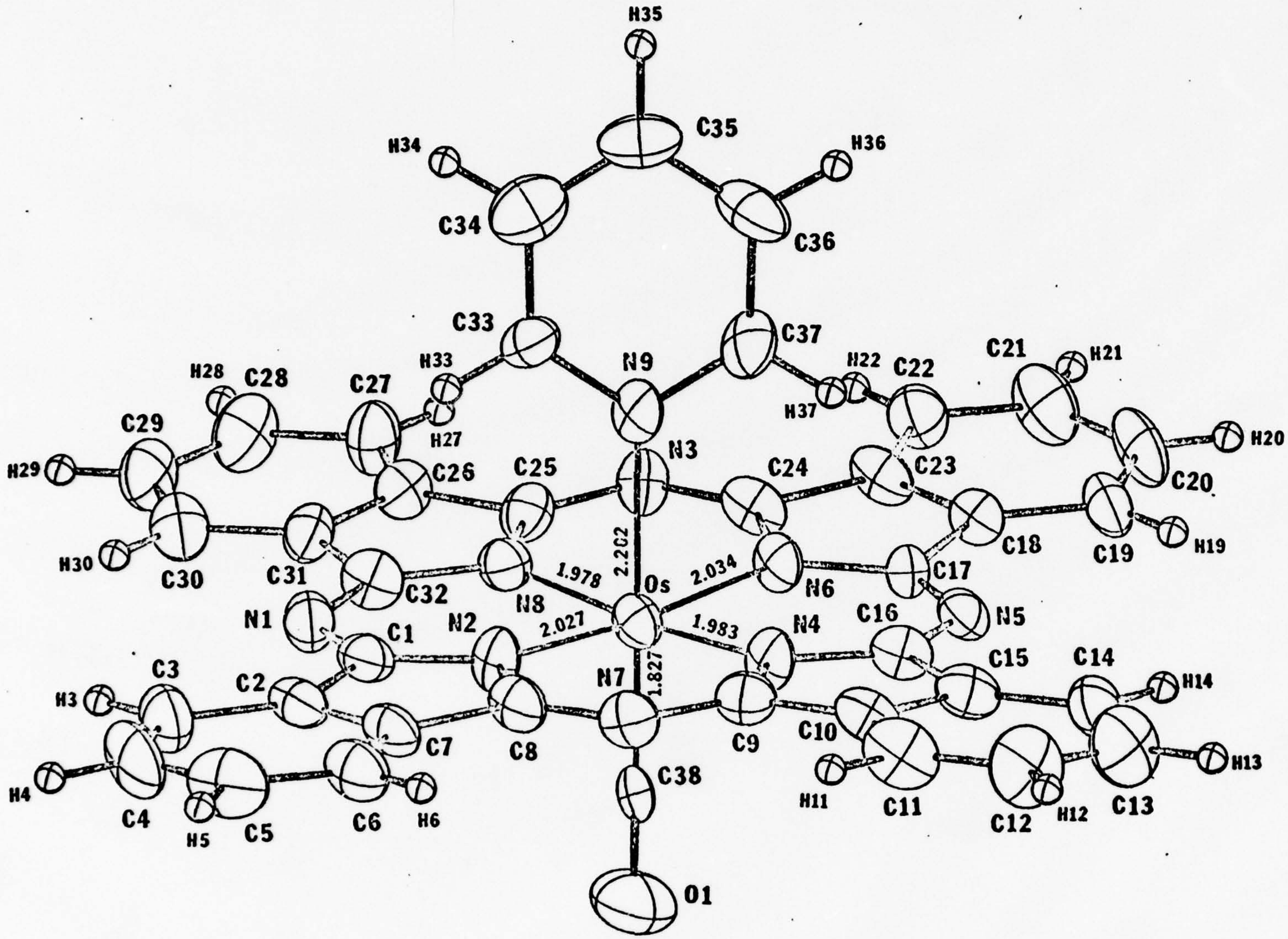
H35
C35
H34
H36
C34
C36
C33
C37
N9
H33
H22
C22
C21
H21
H28
C28
C27
H37
C29
H27
H20
H29
C26
C25
N3
C24
C23
C20
C31
C18
C19
H19
H30
C30
C32
N8
1.978
2.202
Os
2.034
N6
C17
N1
N2
2.027
1.983
N4
C16
N5
C1
C15
C14
C3
N7
1.827
H14
H3
C2
C7
C8
C9
C10
H13
H4
C38
C4
H5
C5
C6
H6
H11
C11
C12
H12
C13
O1

Figure 2. ORTEP[13] drawing of the structure of PcOs(CO)(Py). Numbering scheme is shown. The bond lengths involving the osmium ion are also shown. The thermal ellipsoids are drawn for 50% probability, except those of

Fig. 2 Tsutsui

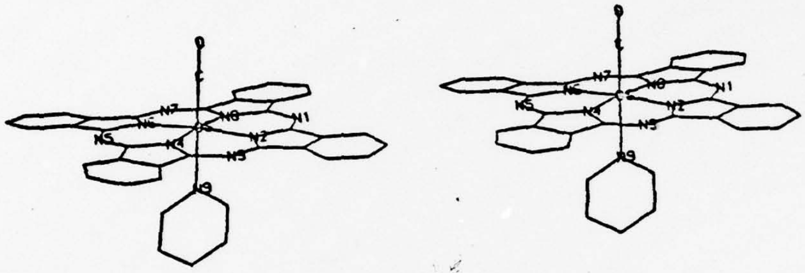
O
C
N7
N8
N1
N6
N5
N4
N2
N3
N9
O
C
N7
N8
N1
N6
N5
N4
N2
N3
N9

Fig. 3 Tsutsui

Figure 3. Stereoview of PcOs(II)(CO)(Py)

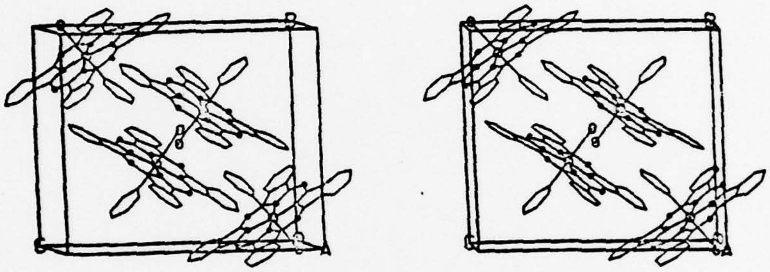

Fig. 4 Tsutsui

Figure 4. Stereoview of the packing in the unit cell. The osmium atoms are indicated by the larger circles, the nitrogen atom. Oxygen atoms of the carbonyl groups are labeled with the letter O.

SYNTHESIS AND STRUCTURE OF A NEW CLASS OF METALLOPHTHALOCYANINES:

Carbonylphthalocyanato(pyridine or THF)-ruthenium(II) and carbonylphthalocyanato (pyridine or THF)osmium(II)

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SUPPLEMENTARY MATERIAL

TABLE III

Calculated Postional Parameters for Hydrogen Atoms

Atom	X	Y	Z
H(3)	0.512	0.197	-0.094
H(4)	0.444	0.292	-0.205
H(5)	0.274	0.368	-0.232
H(6)	0.142	0.342	-0.159
H(11)	-0.145	0.351	-0.064
H(12)	-0.324	0.369	-0.040
H(13)	-0.378	0.292	0.055
H(14)	-0.244	0.198	0.140
H(19)	-0.054	0.028	0.333
H(20)	0.015	-0.053	0.450
H(21)	0.203	-0.108	0.492
H(22)	0.331	-0.083	0.419
H(27)	0.618	-0.090	0.319
H(28)	0.796	-0.117	0.293
H(29)	0.832	-0.055	0.181
H(30)	0.691	0.028	0.093
H(33)	0.449	0.227	0.168
H(34)	0.544	0.327	0.264
H(35)	0.451	0.379	0.356
H(36)	0.256	0.334	0.342
H(37)	0.167	0.233	0.244

TABLE IV

ROOT-MEAN-SQUARE AMPLITUDES OF THERMAL VIBRATION IN ANGSTROMS.

ATOM	MIN.	INT'MED.	MAX.
OS	0.143	0.177	0.190
N1	0.167	0.197	0.203
N2	0.113	0.178	0.219
N3	0.141	0.192	0.217
N4	0.110	0.180	0.239
N5	0.147	0.179	0.197
N6	0.133	0.191	0.200
N7	0.155	0.173	0.218
N8	0.107	0.192	0.241
C1	0.151	0.173	0.247
C2	0.145	0.150	0.248
C3	0.167	0.215	0.251
C4	0.136	0.243	0.299
C5	0.136	0.225	0.330
C6	0.165	0.199	0.254
C7	0.136	0.181	0.230
C8	0.144	0.179	0.229
C9	0.170	0.188	0.205
C10	0.161	0.182	0.232
C11	0.157	0.222	0.271
C12	0.144	0.261	0.289
C13	0.170	0.252	0.272
C14	0.168	0.183	0.232
C15	0.149	0.190	0.225
C16	0.120	0.176	0.211
C17	0.142	0.173	0.201
C18	0.135	0.177	0.195
C19	0.148	0.196	0.234
C20	0.125	0.205	0.292
C21	0.136	0.227	0.287
C22	0.187	0.196	0.232
C23	0.127	0.177	0.242
C24	0.131	0.176	0.235
C25	0.158	0.193	0.202
C26	0.165	0.191	0.201
C27	0.171	0.213	0.248
C28	0.160	0.224	0.268
C29	0.159	0.221	0.256
C30	0.182	0.195	0.236
C31	0.162	0.184	0.208
C32	0.141	0.200	0.215
N9	0.159	0.173	0.212
C33	0.158	0.203	0.213
C34	0.213	0.222	0.274
C35	0.164	0.213	0.283
C36	0.157	0.211	0.293
C37	0.145	0.214	0.239
O1	0.190	0.286	0.326

TABLE V

Values of 10|Fo| and 10 |Fc|

For PcOs(CO)(Py)

```
(PYRIDINE)(CARBONYL)PHTHALOCYANINATOOSMIUM(II)  10 FO AND 10 FC                                                  PAGE  1

 H  K  L   FOBS  FCALC     H  K  L   FOBS  FCALC     H  K  L   FOBS  FCALC     H  K  L   FOBS  FCALC     H  K  L   FOBS  FCALC

 0  0  2    329    347     0  3 17    549    523     0  7  5    467    506     0 12  9    357    415     1  1 -3   2345   2221
 0  0  4   1302   1299     0  4  0    558    437     0  7  6   1085   1204     0 12 11    840    890     1  1 -2   2201   2109
 0  0  6    460    447     0  4  1    283    308     0  7  7    360    373     0 12 12    465    492     1  1 -1   1872   1794
 0  0  8   1881   1850     0  4  2    607    620     0  7  8    495    536     0 13  1    875    872     1  1  0    587    643
 0  0 10   1165   1140     0  4  4   2448   2629     0  7 10   1258   1317     0 13  3    708    715     1  1  1   2298   2413
 0  0 12   1451   1442     0  4  5    586    617     0  7 12    862    902     0 13  5    785    784     1  1  2   1517   1535
 0  0 14    914    872     0  4  6    400    452     0  7 14   1092   1139     0 13  6    368    410     1  1  3    670    681
 0  0 16    598    566     0  4  7    685    723     0  7 16    615    620     0 13  7    386    372     1  1  4    642    612
 0  0 18    529    541     0  4  8   1696   1747     0  8  0   1529   1533     0 13  9    760    788     1  1  5   1391   1385
 0  1  2   2293   2362     0  4 10    758    767     0  8  1    886    869     0 14  0   1104   1096     1  1  6   1448   1466
 0  1  3   1545   1569     0  4 11    346    348     0  8  3    658    704     0 14  2    378    326     1  1  8    347    369
 0  1  4    956    947     0  4 12   1231   1241     0  8  4   1264   1339     0 14  4    835    848     1  1  9    831    897
 0  1  5   1290   1319     0  4 13    401    409     0  8  5    736    801     0 14  8    820    817     1  1 10   1107   1101
 0  1  6   1297   1322     0  4 14    785    822     0  8  7    871    930     0 14 10    513    545     1  1 12    627    612
 0  1  7    953    971     0  4 15    582    602     0  8  8   1076   1131     0 15  1    528    506     1  1 13    847    831
 0  1  8    896    884     0  4 16   1043   1070     0  8 11    846    862     0 15  2    882    886     1  1 14    655    628
 0  1  9   1678   1670     0  5  1   1291   1295     0  8 12    892    929     0 15  5    388    371     1  1 16    782    768
 0  1 10   1357   1339     0  5  3   1573   1652     0  8 14    487    491     0 15  6    790    785     1  1 17    472    492
 0  1 11    433    492     0  5  4    618    629     0  8 15    594    603     0 16  0    424    403     1  2-18    636    612
 0  1 12    543    540     0  5  5   1268   1387     0  9  1   1549   1535     0 16  1    709    687     1  2-15    825    803
 0  1 13   1286   1295     0  5  6    952   1023     0  9  3    857    906     0 16  3    760    755     1  2-14    617    600
 0  1 14    761    756     0  5  7   1057   1130     0  9  5   1644   1754     0 17  1    534    555     1  2-12   1599   1623
 0  1 16    537    540     0  5  9   1205   1268     0  9  7    470    496     1  0-15   1430   1432     1  2-10    636    625
 0  1 17    721    713     0  5 10    509    512     0  9  9   1228   1301     1  0-13    559    595     1  2 -8   2798   2719
 0  2  1   2456   2512     0  5 11    423    431     0  9 13   1177   1231     1  0-11   2034   2011     1  2 -7    688    656
 0  2  2   1895   1979     0  5 12    487    503     0 10  0   1204   1199     1  0 -9   1602   1536     1  2 -5    422    400
 0  2  3   2693   2819     0  5 13   1223   1223     0 10  1    992    965     1  0 -7   1680   1697     1  2 -4   2078   2024
 0  2  4    702    717     0  5 14    370    396     0 10  3    663    679     1  0 -5    466    479     1  2 -3   2070   1934
 0  2  5   1628   1677     0  5 17    930    956     0 10  4   1272   1329     1  0 -3   2196   2168     1  2  0   3424   3521
 0  2  7   1627   1660     0  6  0    889    884     0 10  5    566    587     1  0  1   2428   2390     1  2  1   1830   1885
 0  2  8    415    439     0  6  1   1215   1219     0 10  6    341    334     1  0  3    690    724     1  2  2   1013   1041
 0  2  9    905    916     0  6  2    404    489     0 10  7    767    820     1  0  5    746    728     1  2  3   1372   1398
 0  2 11   1924   1929     0  6  3   1651   1740     0 10  8    769    809     1  0  7   1749   1750     1  2  4    951   1024
 0  2 15   1080   1067     0  6  4    807    855     0 10 11    607    661     1  0 11   1861   1832     1  2  6   1082   1100
 0  3  1   1517   1535     0  6  5    326    372     0 10 12    689    724     1  0 15   1045    996     1  2  8   1606   1621
 0  3  2   3474   3617     0  6  6    979   1014     0 10 14    415    411     1  1-18    384    354     1  2 10    914    909
 0  3  3   1157   1232     0  6  7   1365   1418     0 11  2   1455   1461     1  1-17    616    621     1  2 12   1246   1230
 0  3  4    587    626     0  6  8    668    708     0 11  4    361    365     1  1-14   1087   1084     1  2 14    890    878
 0  3  5   1301   1336     0  6  9    578    524     0 11  5    410    404     1  1-13   1003   1016     1  2 16    673    634
 0  3  6   1717   1816     0  6 10    371    372     0 11  6   1256   1332     1  1-12    658    636     1  2 17    481    467
 0  3  8    753    769     0  6 11   1185   1247     0 11 10    742    750     1  1-11    427    488     1  3-18    386    387
 0  3  9    999   1074     0  6 12    570    612     0 11 12    618    659     1  1-10   1092   1053     1  3-17    837    847
 0  3 10   1447   1438     0  6 14    590    636     0 12  1    756    735     1  1 -9   1352   1339     1  3-16    304    292
 0  3 12    467    408     0  6 15   1128   1172     0 12  3   1222   1205     1  1 -8    712    673     1  3-14    638    634
 0  3 13    839    844     0  7  1    288    275     0 12  4    523    540     1  1 -7   1169   1159     1  3-13   1129   1115
 0  3 14    742    735     0  7  2   2099   2178     0 12  5    481    520     1  1 -6   1362   1370     1  3-12    344    327
 0  3 16    892    806     0  7  4    440    412     0 12  7    694    723     1  1 -5    829    815     1  3-11    831    812
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(PYRIDINE)(CARBONYL)PHTALOCYANATOOSMIUM(II) 10 FO AND 10 FC                                                   PAGE  2

 H  K  L   FOBS  FCALC     H  K  L   FOBS  FCALC     H  K  L   FOBS  FCALC     H  K  L   FOBS  FCALC     H  K  L   FOBS  FCALC

 1  3-10    904    877     1  4 15   1100   1090     1  6  8   1549   1599     1  8  9    340    322     1 11  4    371    388
 1  3 -9   1649   1578     1  5-16    697    686     1  6 10    787    834     1  8 10    365    363     1 11  5   1293   1371
 1  3 -7    936    906     1  5-14    637    607     1  6 11    566    583     1  8 11    616    603     1 11  7    566    595
 1  3 -6   1291   1229     1  5-13    425    414     1  6 12    529    502     1  8 12    477    499     1 11  9    613    864
 1  3 -5   2323   2230     1  5-10   1936   1898     1  6 14    775    775     1  8 14    502    533     1 11 13    821    899
 1  3 -4   1371   1292     1  5 -9    700    682     1  6 16    562    564     1  8 15    881    903     1 12-12    828    814
 1  3 -3    320    336     1  5 -8    545    530     1  7-14    386    377     1  9-14   1219   1204     1 12 11    599    595
 1  3 -2   1360   1303     1  5 -7    976    941     1  7-13   1372   1366     1  9-12    712    719     1 12-10    437    428
 1  3 -1   1577   1532     1  5 -6   2329   2308     1  7-11    496    471     1  9-10   1145   1137     1 12 -9    415    419
 1  3  0   1026    989     1  5 -5    422    420     1  7 -9   1531   1517     1  9 -6   1368   1374     1 12 -8    956    963
 1  3  1   2311   2381     1  5 -3    727    714     1  7 -7   1072   1078     1  9 -2   1603   1622     1 12 -7    516    507
 1  3  2   3427   3603     1  5 -2   2084   2052     1  7 -6    445    433     1  9  2   1300   1348     1 12 -6    341    298
 1  3  3   2418   2524     1  5  2   2609   2670     1  7 -5   1252   1246     1  9  4    903    917     1 12 -5    557    539
 1  3  4    414    393     1  5  3    521    503     1  7 -4    301    250     1  9  6   1715   1815     1 12 -4   1196   1154
 1  3  5   2048   2118     1  5  4    317    307     1  7 -3   1181   1172     1  9  8    663    709     1 12 -3    435    444
 1  3  6    361    421     1  5  5   1241   1294     1  7 -2    337    327     1  9 10    861    894     1 12 -1    358    361
 1  3  7    379    413     1  5  6   1802   1905     1  7 -1   1355   1344     1  9 12    694    714     1 12  0   1011   1006
 1  3  8    538    563     1  5  7    487    498     1  7  1   1870   1924     1  9 14    773    781     1 12  1    440    439
 1  3  9   1351   1379     1  5  8   1075   1128     1  7  3    620    647     1 10-15    686    683     1 12  2    544    484
 1  3 10    536    544     1  5  9    549    581     1  7  4   1445   1457     1 10-12   1143   1157     1 12  3    752    757
 1  3 12    570    584     1  5 10   1251   1307     1  7  5   1655   1762     1 10-11   1088   1097     1 12  4   1126   1171
 1  3 13   1327   1312     1  5 12    816    836     1  7  6    747    772     1 10 -9    408    421     1 12  5    504    525
 1  3 14    440    436     1  5 13    485    460     1  7  7    618    655     1 10 -8    685    677     1 12  6    711    733
 1  3 17    938    917     1  5 14    650    655     1  7  9   1582   1655     1 10 -7    941    893     1 12  7    677    720
 1  4-15    790    766     1  5 16    772    753     1  7 12    367    400     1 10 -5    895    879     1 12  8    788    843
 1  4-12    504    484     1  6-16    729    718     1  7 13    972    965     1 10 -4   1320   1314     1 12 10    511    559
 1  4-11   1736   1695     1  6-15    779    768     1  8-16    495    478     1 10 -3    724    722     1 12 11    484    530
 1  4-10    324    274     1  6-14    537    528     1  8-15    995    986     1 10 -1    704    709     1 12 12    436    446
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 1  4 -7   1776   1747     1  6-11    433    418     1  8-12    890    895     1 10  3   1036   1067     1 13 -5    352    352
 1  4 -6    822    792     1  6-10    498    493     1  8-11    900    883     1 10  4    981   1020     1 13 -2   1258   1231
 1  4 -5   1559   1547     1  6 -9    395    388     1  8 -9    463    473     1 10  6    360    410     1 13  0    340    347
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(PYRIDINE)(CARBONYL)PHTALOCYANATOOSMIUM(II) 10 FO AND 10 FC PAGE 3

H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC
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1	14	3	1072	1091	2	1	-2	1652	1605	2	3	-7	1217	1161	2	4	16	471	475	2	6	14	368	392
1	14	4	768	767	2	1	-1	263	245	2	3	-6	2321	2251	2	5	-17	1023	1031	2	6	15	944	972
1	14	7	984	1043	2	1	0	912	938	2	3	-5	544	548	2	5	-13	983	963	2	7	-14	1147	1133
1	15	-7	471	445	2	1	1	1984	2043	2	3	-4	315	294	2	5	-11	1105	1065	2	7	-12	425	441
1	15	-6	432	419	2	1	2	879	897	2	3	-3	557	561	2	5	-10	708	706	2	7	-10	1611	1627
1	15	-5	668	646	2	1	4	710	747	2	3	-2	1269	1240	2	5	-9	1675	1653	2	7	-6	2076	2023
1	15	-3	738	700	2	1	5	1560	1638	2	3	-1	241	238	2	5	-7	1606	1550	2	7	-3	491	479
1	15	-2	530	515	2	1	6	1074	1081	2	3	0	813	810	2	5	-6	999	986	2	7	-2	2366	2403
1	15	-1	753	773	2	1	7	504	492	2	3	1	332	369	2	5	-5	1934	1870	2	7	0	735	762
1	15	1	736	749	2	1	8	1273	1244	2	3	2	2401	2502	2	5	-4	486	464	2	7	1	492	484
1	15	2	489	498	2	1	9	1090	1084	2	3	3	623	622	2	5	-3	1354	1333	2	7	2	1558	1647
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1	15	4	373	358	2	1	12	673	664	2	3	5	915	993	2	5	-1	839	836	2	7	4	385	465
1	15	5	970	1020	2	1	13	1134	1099	2	3	6	1149	1214	2	5	0	444	452	2	7	5	1459	1444
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1	16	0	983	1001	2	1	16	632	608	2	3	8	712	734	2	5	2	400	427	2	7	7	468	470
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2	0	-16	1458	1455	2	2	-13	619	623	2	3	13	623	601	2	5	6	275	302	2	7	14	526	542
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(PYRIDINE)(CARBONYL)PHTALOCYANATOOSMIUM(II) 10 FO AND 10 FC                                       PAGE  4

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 2  9-13    1051   1027     2 12-12     735    765     2 15  4     518    547     3  1 12     964    903     3  3  6     578    617
 2  9-11     600    587     2 12-11     951    941     2 15  5     435    457     3  1 13     839    794     3  3  8     556    562
 2  9 -9    1026   1012     2 12 -9     753    764     2 15  6     692    684     3  1 14     399    378     3  3  9    1544   1556
 2  9 -7    1431   1378     2 12 -8     462    400     2 16 -5     727    675     3  1 16     566    525     3  3 10     609    619
 2  9 -5    1157   1105     2 12 -7     717    686     2 16 -3     579    571     3  2-18     390    409     3  3 11     708    692
 2  9 -3    1974   1891     2 12 -5    1007    969     2 16 -1     810    831     3  2-16    1402   1436     3  3 12     475    461
 2  9 -1    1306   1296     2 12 -4     983    959     2 16  1     416    383     3  2-12    1352   1362     3  3 13    1183   1172
 2  9  1    1435   1456     2 12 -3     756    748     2 16  3     889    908     3  2 -8    2104   2102     3  4-17     529    537
 2  9  3     399    435     2 12 -1    1187   1115     3  0-15    1566   1578     3  2 -7    1115   1075     3  4-16     454    481
 2  9  5    2145   2216     2 12  0     548    633     3  0-13     548    588     3  2 -6     796    769     3  4-15     690    883
 2  9  7     533    533     2 12  1     362    403     3  0-11    1276   1294     3  2 -4    2680   2608     3  4-13    1266   1246
 2  9  9    1533   1635     2 12  3    1145   1174     3  0 -9    2470   2456     3  2 -3     467    414     3  4-12     662    659
 2  9 13     954    958     2 12  4     696    722     3  0 -7    1441   1444     3  2 -2     907    876     3  4-11    1307   1303
 2 10-15     640    643     2 12  7    1165   1217     3  0 -5    2268   2287     3  2 -1     563    562     3  4 -9     726    740
 2 10-14     370    382     2 12  8     593    619     3  0 -3    1309   1293     3  2  0    1903   2028     3  4 -8     643    650
 2 10-13     504    512     2 12  9     412    437     3  0 -1    1989   2009     3  2  1     711    709     3  4 -7    1296   1279
 2 10-12     886    876     2 12 10     511    540     3  0  3    2275   2283     3  2  2    1045   1099     3  4 -5    2331   2207
 2 10-11     697    696     2 12 11     679    683     3  0  7    1597   1588     3  2  3     421    458     3  4 -4     790    780
 2 10 -8    1015   1012     2 13-11     765    536     3  0 11    1717   1617     3  2  4     730    838     3  4 -3    1170   1145
 2 10 -7     868    859     2 13 -9     996    926     3  0 13     345    322     3  2  5     757    765     3  4 -2     400    403
 2 10 -5     875    862     2 13 -7     726    680     3  0 15     727    704     3  2  6    1125   1168     3  4 -1    1575   1607
 2 10 -4    1440   1409     2 13 -6     433    452     3  1-18     878    919     3  2  8    1172   1192     3  4  0     504    517
 2 10 -3     751    676     2 13 -5     832    794     3  1-17     673    723     3  2  9     453    455     3  4  1     575    582
 2 10 -1     929    898     2 13 -3    1054   1014     3  1-15     649    677     3  2 10    1283   1249     3  4  2     537    571
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 2 10  1     657    643     2 13  1    1130   1157     3  1-13     818    822     3  2 12     864    843     3  4  4     774    785
 2 10  2     871    853     2 13  3     457    481     3  1-11     405    421     3  2 14    1029    962     3  4  6     471    492
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 2 10  4    1038   1091     2 13  5    1113   1134     3  1 -9     982    969     3  3-17     903   1020     3  4 10     532    547
 2 10  6     434    474     2 13  9     913    923     3  1 -8     339    325     3  3-15     579    594     3  4 11    1417   1390
 2 10  7    1797   1896     2 14 -9     690    692     3  1 -7    1867   1830     3  3-14     852    865     3  4 14     374    380
 2 10  8     840    879     2 14 -8    1140   1096     3  1 -6    1923   1895     3  3-13    1321   1317     3  4 15     808    782
 2 10 10     642    685     2 14 -7     398    412     3  1 -5     931    908     3  3-11    1157   1137     3  5-18     947    950
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 2 10 12     551    546     2 14 -1     370    403     3  1 -2    2163   2071     3  3 -9    1081   1073     3  5-16     442    424
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 2 11-10    1152   1132     2 14  2     417    409     3  1  0     899    920     3  3 -6    1368   1342     3  5-13     504    522
 2 11 -6    1447   1428     2 14  3     451    466     3  1  1    1009   1049     3  3 -5    1234   1200     3  5-11     718    711
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 2 11  2    1541   1567     2 14  8     669    697     3  1  4     693    717     3  3 -2    1058   1021     3  5 -6    2260   2214
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 2 11  8     770    807     2 15 -2     712    705     3  1  8     753    774     3  3  3     494    507     3  5 -2    1626   1706
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(PYRIDINE)(CARBONYL)PHTHALOCYANATOOSMIUM(II) 10 FO AND 10 FC · PAGE 5

H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC
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3	5	4	1064	1104	3	7	-1	578	570	3	10	-9	399	372	3	12	10	645	635	4	1	-14	1143	1154
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3	5	6	1132	1200	3	7	2	618	621	3	10	-7	413	417	3	13	-7	373	386	4	1	-11	1145	1167
3	5	7	307	293	3	7	5	2118	2164	3	10	-5	1291	1239	3	13	-6	977	939	4	1	-10	1128	1111
3	5	8	1381	1409	3	7	6	326	329	3	10	-4	1392	1326	3	13	-4	373	355	4	1	-9	798	788
3	5	9	678	676	3	7	9	1551	1641	3	10	-3	691	702	3	13	-3	384	341	4	1	-8	956	922
3	5	10	946	977	3	7	10	401	431	3	10	-2	596	593	3	13	-2	1231	1195	4	1	-7	746	778
3	5	12	1127	1154	3	7	13	700	681	3	10	-1	1249	1230	3	13	0	610	621	4	1	-6	1276	1256
3	5	13	388	383	3	8	-16	613	605	3	10	0	1055	1058	3	13	2	1056	1068	4	1	-5	505	531
3	6	-16	857	836	3	8	-15	791	790	3	10	1	431	451	3	13	4	595	616	4	1	-4	684	640
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(PYRIDINE)(CARBONYL)PHTHALOCYANATOOSMIUM(II)  10 FO AND 10 FC                                                    PAGE  6

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 4  2  9    400    383     4  4 11    424    441     4  6  7   1202   1226     4  8 11    551    561     4 12-11    438    429
 4  2 10    406    366     4  4 12    465    501     4  6  8    341    359     4  9-15    714    699     4 12 -9    859    857
 4  2 11   1507   1453     4  4 14    855    911     4  6 10    633    686     4  9-13    932    949     4 12 -8    524    536
 4  2 13    535    405     4  5-17    861    912     4  6 11    835    849     4  9-11    624    607     4 12 -7    706    689
 4  2 15    556    827     4  5-15    913    929     4  6 13    427    423     4  9 -9   1057   1045     4 12 -5    916    876
 4  3-18    753    803     4  5-14    530    570     4  6 14    390    368     4  9 -7   1505   1433     4 12 -4    774    776
 4  3-17    373    400     4  5-13    966    984     4  7-14   1352   1420     4  9 -5    342    315     4 12 -2    463    457
 4  3-14   1273   1286     4  5-12    384    384     4  7-11    565    571     4  9 -3   1677   1633     4 12 -1   1151   1130
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 4  3 -7   1566   1520     4  5 -6    277    268     4  7 -3    546    515     4 10-15    432    474     4 13-11    836    807
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 4  3 -4    813    790     4  5 -4    568    562     4  7  0   1263   1258     4 10-12    875    894     4 13 -7    746    716
 4  3 -3   1066   1026     4  5 -3   2240   2160     4  7  2   1289   1318     4 10-11    383    383     4 13 -6    425    417
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 4  3  6    998   1019     4  5  9   1106   1153     4  8-16    773    798     4 10 -1   1140   1107     4 14  0    927    950
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 4  3  9    659    662     4  5 11    640    633     4  8-13    475    480     4 10  2    651    672     4 14  4    616    620
 4  3 12   1214   1201     4  5 13    682    633     4  8-12    611    602     4 10  3    831    878     4 14  6    560    528
 4  3 13    666    643     4  6-17    518    515     4  8-11    702    697     4 10  4    481    474     4 15 -7    498    515
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 4  4-13    347    348     4  6-15    844    875     4  8 -9    625    627     4 10  7    860    843     4 15 -2    571    598
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(PYRIDINE)([illegible])PHTHALOCYANINATOOSMIUM(II) 10 FO AND 10 FC PAGE 7

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5	1	-13	611	627
5	1	-12	362	380
5	1	-11	1018	1040
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5	1	-9	595	617
5	1	-8	375	391
5	1	-7	1190	1211
5	1	-6	1067	1112
5	1	-4	621	653
5	1	-3	1116	1096
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5	12	-13	492	477
5	12	-12	831	843
5	12	-9	391	376
5	12	-8	999	958
5	12	-5	450	419
5	12	-4	712	670
5	12	-2	761	753
5	12	-1	688	690
5	12	0	817	823
5	12	2	719	741
5	12	3	765	786
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5	12	6	805	817
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H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCAL
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5	13	-4	675	657	6	1	4	1140	1107	6	3	7	614	596	6	5	11	562	517	6	8	-6	890	89
5	13	-2	536	509	6	1	5	1262	1217	6	3	8	1103	1078	6	6	-17	744	797	6	8	-5	1170	11
5	13	0	632	612	6	1	6	408	390	6	3	9	495	462	6	6	-16	535	546	6	8	-4	830	81
5	13	1	370	391	6	1	7	534	497	6	3	11	430	426	6	6	-15	543	545	6	8	-3	486	48
5	13	2	846	866	6	1	8	915	887	6	3	12	708	671	6	6	-13	1138	1166	6	8	-2	897	89
5	13	4	772	792	6	1	9	805	752	6	4	-16	1048	1108	6	6	-11	558	534	6	8	-1	817	87
5	13	6	445	441	6	1	12	528	470	6	4	-13	518	503	6	6	-10	563	558	6	8	0	801	83
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5	14	-6	414	443	6	2	-15	459	466	6	4	-9	717	695	6	6	-7	360	349	6	8	5	449	46
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6	1	-4	[illegible]	519	6	3	0	1073	1175	6	5	2	511	309	6	8	-16	736	719	6	11	-10	877	91
6	1	-3	1556	1529	6	3	1	874	888	6	5	3	474	497	6	8	-15	416	436	6	11	-8	758	73
6	1	-2	1130	1141	6	3	2	1023	1029	6	5	4	739	772	6	8	-13	670	688	6	11	-6	1120	109
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6	1	1	1506	1541	6	3	5	819	830	6	5	8	467	413	6	8	-9	768	765	6	11	0	734	73

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(PYRIDINE)(CARBONYL)PHTHALOCYANINATOOSMIUM(II), 10 FO AND 10 FC                                                    · PAGE  9

H  K  L   FOBS  FCALC    H  K  L   FOBS  FCALC    H  K  L   FOBS  FCALC    H  K  L   FOBS  FCALC    H  K  L   FOBS  FCALC

6 11  1    459    459    7  0  9    697    661    7  3 -8    564    547    7 [illegible]      411    7  8 -1   1291   1325
6 11  2    352    367    7  0 11    626    578    7  3 -7   1668   1626    7  5  4 [illegible] 1083   7  8  2    581    573
6 11  4   1174   1274    7  1-18    707    829    7  3 -6    369    358    7  5  8   1296   1298    7  8  5    534    567
6 11  8    834    769    7  1-17    437    473    7  3 -4    550    588    7  6-16    368    682    7  8  6    805    833
6 12-11    561    576    7  1-16    433    466    7  3 -3   1503   1521    7  6-15    372    418    7  8  7    890    864
6 12-10    451    450    7  1-15    702    719    7  3 -2    636    646    7  6-14    582    621    7  8  9    522    487
6 12 -9    929    938    7  1-14    732    724    7  3 -1    463    508    7  6-13    534    555    7  9-14    978   1009
6 12 -8    481    527    7  1-13    402    407    7  3  0    980   1022    7  6-12   1034    985    7  9-12    511    536
6 12 -6    619    614    7  1-12    628    625    7  3  1   1179   1253    7  6-10    506    472    7  9-10    645    643
6 12 -5   1198   1164    7  1-11    952    982    7  3  3    471    459    7  6 -9    736    721    7  9 -8    857    845
6 12 -4    742    739    7  1-10   1207   1217    7  3  4    483    517    7  6 -8    929    912    7  9 -6   1149   1128
6 12 -2    402    374    7  1 -8   1182   1156    7  3  5    915    928    7  6 -6    935    918    7  9 -4   1391   1367
6 12 -1    914    920    7  1 -7   1722   1672    7  3  7    406    408    7  6 -5    785    774    7  9 -2    859    890
6 12  2    557    573    7  1 -6   1329   1303    7  3  8    603    575    7  6 -4    890    913    7  9  0    727    750
6 12  3    843    896    7  1 -4   1176   1143    7  3  9    712    690    7  6 -2    843    906    7  9  4   1089   1120
6 12  4    445    515    7  1 -3   1291   1342    7  3 11    676    633    7  6 -1    630    644    7  9  8    959    922
6 12  5    371    344    7  1 -2    641    690    7  4-17    948    968    7  6  0    750    772    7 10-13    732    735
6 12  6    514    465    7  1  0   1211   1230    7  4-13    920    897    7  6  1    614    624    7 10-12    672    716
6 12  7    623    623    7  1  1    746    780    7  4-11    344    293    7  6  2   1169   1225    7 10 -9    714    712
6 13-10    394    376    7  1  2    471    471    7  4-10    608    592    7  6  3    382    417    7 10 -8    598    594
6 13 -7   1247   1257    7  1  3    422    431    7  4 -9   1455   1421    7  6  6    976    959    7 10 -6    778    757
6 13 -4    407    414    7  1  4   1084   1043    7  4 -5   1803   1781    7  6  7    488    507    7 10 -5   1097   1093
6 13 -3    828    817    7  1  5    769    762    7  4 -2    702    727    7  6 10    843    836    7 10 -4    769    770
6 13 -2    353    346    7  1  7    696    675    7  4 -1   1296   1358    7  7-15   1108   1144    7 10 -2    762    758
6 13  1   1068   1038    7  1  8    642    606    7  4  0    372    411    7  7-14    405    415    7 10 -1    734    718
6 13  5    757    761    7  1  9    534    487    7  4  1    624    653    7  7-11    919    912    7 10  2    527    534
6 14 -8    736    742    7  1 11    431    364    7  4  2    460    452    7  7-10    380    369    7 10  3    668    678
6 14 -6    526    509    7  1 12    499    431    7  4  3   1633   1677    7  7 -7   1435   1403    7 10  5    535    526
6 14 -5    552    563    7  2-16   1156   1216    7  4  5    512    496    7  7 -3   1648   1649    7 10  6    652    610
6 14 -4    549    531    7  2-14    386    365    7  4  7    678    663    7  7 -2    397    417    7 10  7    551    532
6 14 -2    584    555    7  2-12    949    952    7  4  9    741    703    7  7 -1    576    607    7 11-11   1104   1162
6 14  0    617    620    7  2-10   1153   1157    7  4 10    390    351    7  7  1   1208   1243    7 11 -7   1292   1291
6 14  2    849    821    7  2 -8   1594   1585    7  4 11    595    545    7  7  3    496    486    7 11 -3   1107   1089
6 15 -4    466    478    7  2 -6   1627   1611    7  5-14    917    968    7  7  5   1024   1003    7 11  1    687    662
6 15 -3    501    532    7  2 -5    410    447    7  5-12    865    855    7  7  7    795    807    7 11  3    407    404
7  0-17    971    992    7  2 -4   1129   1117    7  5-11    529    495    7  7  9    753    745    7 11  5    799    793
7  0-15    430    448    7  2 -2   1511   1617    7  5-10    883    854    7  8-15    399    405    7 12 -9    549    590
7  0-13   1179   1209    7  2  0    734    775    7  5 -9    349    345    7  8-14    451    445    7 12 -8    532    501
7  0-11    363    380    7  2  1    405    356    7  5 -8    932    943    7  8-13    997   1030    7 12 -6    685    670
7  0 -9 [illegible] [illegible] 7  2  2   1221   1256    7  5 -7    419    417    7  8-12    645    685    7 12 -5    536    535
7  0 -5   2521   2519    7  2  6   1309   1323    7  5 -6    815    801    7  8-10    675    661    7 12 -4    584    624
7  0 -3    451    494    7  2 10    964    884    7  5 -4   1412   1408    7  8 -9    958    924    7 12 -2    694    679
7  0 -1   1595   1607    7  3-18    627    680    7  5 -3    400    416    7  8 -8    638    615    7 12  0    497    517
7  0  1    479    477    7  3-15    795    827    7  5 -2    623    649    7  8 -6    688    684    7 12  2    689    683
7  0  3    929    937    7  3-14    470    508    7  5  0   1245   1322    7  8 -5   1113   1117    7 12  3    602    590
7  0  5    818    799    7  3-11   1197   1172    7  5  1    543    660    7  8 -4    412    437    7 13 -8    525    575
7  0  7   1121   1099    7  3-10    834    802    7  5  2    619    637    7  8 -2    944    970    7 13 -6    752    716
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(PYRIDINE)(CARBONYL)PHTALOCYANATOOSMIUM(II)  10 FO AND 10 FC                                                   PAGE 10

 H  K  L   FOBS  FCALC      H  K  L   FOBS  FCALC      H  K  L   FOBS  FCALC      H  K  L   FOBS  FCALC      H  K  L   FOBS  FCALC

 7 13 -4    723    667      8  2 -2    377    411      8  5-15   1061   1111      8  8-12    397    390      8 13 -3    965    922
 7 13 -2    393    336      8  2 -1   1226   1310      8  5-14    439    453      8  8-10    531    527      9  0-17    925    943
 7 13  0    769    771      8  2  1    727    755      8  5-11   1245   1254      8  8 -9    581    587      9  0-13    777    793
 7 14 -5    914    843      8  2  3   1247   1238      8  5 -8    525    519      8  8 -6    812    804      9  0 -9   2373   2472
 7 14 -2    379    365      8  2  5    758    735      8  5 -7   1356   1332      8  8 -5    732    718      9  0 -5   1504   1490
 7 14 -1    812    770      8  2  7    590    537      8  5 -4    678    677      8  8 -4    449    442      9  0 -3    516    552
 8  0-16   1248   1390      8  2  9    773    694      8  5 -3   1648   1675      8  8 -2    970    980      9  0 -1   1037   1006
 8  0-14    746    746      8  2 10    395    419      8  5 -2    453    441      8  8 -1    700    743      9  0  1   1124   1087
 8  0-12    543    538      8  3-16    384    371      8  5 -1    776    784      8  8  2    635    623      9  0  3   1410   1352
 8  0-10    936    971      8  3-15    417    447      8  5  0    559    564      8  8  3    592    583      9  0  5    997    922
 8  0 -8   1520   1536      8  3-14    966   1007      8  5  1   1266   1308      8  8  6    916    863      9  0  9    521    432
 8  0 -6   1673   1660      8  3-12    992   1010      8  5  3    400    358      8  9-11    925    931      9  1-16    528    548
 8  0 -4    478    535      8  3-11   1088   1101      8  5  4    370    357      8  9 -7    857    842      9  1-15    750    810
 8  0 -2   1362   1393      8  3-10   1016   1015      8  5  5    435    416      8  9 -3   1032   1017      9  1-14    680    706
 8  0  0    510    504      8  3 -8    907    892      8  5  7    644    605      8  9 -1    449    418      9  1-12    761    785
 8  0  2   1279   1261      8  3 -7    780    784      8  5  8    369    323      8  9  1    647    635      9  1-11    843    850
 8  0  6   1445   1342      8  3 -6    900    862      8  5  9    565    558      8  9  3    613    608      9  1-10    751    793
 8  0 10    826    760      8  3 -5    405    405      8  6-16    566    561      8  9  5    831    813      9  1 -9    388    402
 8  1-16    418    451      8  3 -4   1391   1370      8  6-13   1275   1207      8  9  7    634    534      9  1 -8   1153   1172
 8  1-15   1087   1162      8  3 -3    957    974      8  6-10    488    501      8 10-13    398    400      9  1 -7    984    981
 8  1-14    439    429      8  3 -2    411    439      8  6 -9   1050   1044      8 10-12    623    621      9  1 -4    854    899
 8  1-13    390    448      8  3  0   1415   1518      8  6 -8    371    331      8 10-10    529    529      9  1 -3    550    569
 8  1-12    354    361      8  3  1    801    861      8  6 -6    396    378      8 10 -9    396    380      9  1 -1    505    501
 8  1-11    843    849      8  3  4    957    958      8  6 -5   1203   1271      8 10 -6    504    487      9  1  0   1147   1135
 8  1-10    715    750      8  3  5    386    347      8  6 -4    603    550      8 10 -5    534    517      9  1  1    772    762
 8  1 -8   1128   1134      8  3  7    423    412      8  6 -3    605    602      8 10 -4    426    458      9  1  3    753    718
 8  1 -7   1585   1559      8  3  8    846    811      8  6 -2    850    845      8 10 -2    736    735      9  1  4   1107   1005
 8  1 -6    802    783      8  3  9    405    457      8  6 -1   1395   1437      8 10 -1    747    735      9  1  5    476    443
 8  1 -5    325    302      8  4-16    748    758      8  6  1    570    582      8 10  2    673    670      9  1  7    550    479
 8  1 -4    923    956      8  4-14    793    817      8  6  3    583    557      8 10  3    521    501      9  1  8    510    414
 8  1 -3    776    904      8  4-13    543    541      8  6  5    594    577      8 11-10    564    610      9  2-16    898    942
 8  1 -2    599    634      8  4-12   1350   1375      8  6  6    638    644      8 11 -8    452    435      9  2-14    860    890
 8  1 -1    486    511      8  4-10    956    950      8  6  7    583    547      8 11 -6    441    439      9  2-12    757    760
 8  1  0    951    972      8  4 -9    487    484      8  6  9    590    564      8 11 -4    890    852      9  2-10   1163   1220
 8  1  1    978    981      8  4 -8    900    888      8  7-14    877    912      8 11  0    934    923      9  2 -8    709    660
 8  1  3    831    837      8  4 -6   1161   1152      8  7-12    682    709      8 11  4    807    786      9  2 -6   1593   1572
 8  1  4    718    721      8  4 -5    556    521      8  7-10    620    584      8 12-10    411    421      9  2 -4    355    331
 8  1  5    861    815      8  4 -4    724    755      8  7 -8    758    754      8 12 -9    633    622      9  2 -2   1131   1203
 8  1  7    413    361      8  4 -2   1582   1542      8  7 -6    714    713      8 12 -6    410    408      9  2  2   1276   1277
 8  1  9    756    689      8  4 -1    391    410      8  7 -4   1290   1300      8 12 -5    786    774      9  2  5    354    348
 8  2-17   1156   1238      8  4  0    579    610      8  7 -3    302    411      8 12 -4    400    398      9  2  6    977    878
 8  2-13   1301   1345      8  4  1    583    596      8  7 -2    622    636      8 12 -3    543    515      9  3-15    992   1032
 8  2-10    361    363      8  4  2   1472   1502      8  7  0    954   1017      8 12 -2    435    390      9  3-12    904    602
 8  2 -9   1640   1647      8  4  3    393    380      8  7  4   1042   1000      8 12 -1    741    736      9  3-11    979   1000
 8  2 -7    346    318      8  4  6    608    579      8  7  8    772    714      8 12  2    412    424      9  3-10    624    659
 8  2 -5   1773   1787      8  4  9    406    392      8  8-14    457    459      8 13 -7    668    653      9  3 -8    543    540
 8  2 -3    464    483      8  4 10    860    804      8  8-13    621    616      8 13 -4    375    355      9  3 -7   1346   1310
```

(PYRIDINE)(CARBONYL)PHTHALOCYANATOOSMIUM(II) 10 FO AND 10 FC

H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC
9	3	-4	635	609	9	6	-1	515	553	9	11	-1	530	517	10	3	-7	696	653	10	7	-4	1074	1048
9	3	-3	1121	1145	9	6	1	582	582	9	11	1	699	719	10	3	-6	403	386	10	7	0	1019	991
9	3	-1	676	706	9	6	2	1019	1010	9	12	-6	742	726	10	3	-4	860	883	10	7	4	828	822
9	3	0	665	679	9	6	5	394	363	9	12	-5	425	385	10	3	-1	405	423	10	8	-12	422	414
9	3	1	904	932	9	6	6	711	683	9	12	-2	734	724	10	3	0	772	762	10	8	-10	1120	1135
9	3	3	555	528	9	7	-11	1134	1134	10	0	-16	553	603	10	3	1	427	415	10	8	-9	690	695
9	3	4	530	549	9	7	-7	985	950	10	0	-14	782	892	10	3	3	465	445	10	8	-7	439	411
9	3	5	555	526	9	7	-3	1009	1003	10	0	-12	430	450	10	3	4	1022	959	10	8	-6	549	523
9	3	7	608	537	9	7	-1	675	688	10	0	-10	1311	1327	10	4	-14	785	849	10	8	-5	518	511
9	3	8	483	406	9	7	1	869	860	10	0	-8	761	765	10	4	-12	514	534	10	8	-2	875	860
9	4	-13	1500	1566	9	7	3	778	751	10	0	-6	611	579	10	4	-10	713	710	10	8	-1	473	488
9	4	-12	464	501	9	7	5	641	617	10	0	-2	637	607	10	4	-9	459	494	10	8	1	545	525
9	4	-10	448	483	9	8	-13	727	732	10	0	2	961	871	10	4	-6	1618	1618	10	8	2	646	630
9	4	-9	523	471	9	8	-12	579	581	10	0	6	920	825	10	4	-5	473	453	10	9	-11	1055	1056
9	4	-7	469	457	9	8	-10	564	580	10	1	-16	414	458	10	4	-2	1002	1016	10	9	-7	726	702
9	4	-5	1142	1112	9	8	-9	1113	1141	10	1	-15	736	732	10	4	1	467	475	10	9	-5	573	545
9	4	-4	341	335	9	8	-8	490	501	10	1	-12	610	619	10	4	2	1087	1076	10	9	-3	808	807
9	4	-3	694	668	9	8	-6	377	344	10	1	-11	887	937	10	4	6	881	809	10	9	-1	582	570
9	4	-2	392	372	9	8	-5	603	568	10	1	-10	377	370	10	5	-11	942	950	10	9	1	655	654
9	4	-1	830	863	9	8	-4	400	366	10	1	-9	453	508	10	5	-9	402	367	10	10	-9	818	819
9	4	1	718	782	9	8	-3	507	516	10	1	-8	938	951	10	5	-8	426	403	10	10	-8	490	507
9	4	3	673	692	9	8	-2	746	768	10	1	-7	668	637	10	5	-7	1233	1240	10	10	-6	612	603
9	4	5	651	635	9	8	-1	1021	991	10	1	-5	617	640	10	5	-5	447	404	10	10	-5	418	377
9	4	6	377	335	9	8	1	472	494	10	1	-4	686	743	10	5	-4	478	506	10	10	-2	637	598
9	4	7	538	477	9	8	2	676	668	10	1	-3	531	548	10	5	-3	766	761	10	10	-1	477	477
9	5	-15	434	451	9	8	3	478	419	10	1	-1	385	362	10	5	-1	811	832	11	0	-13	783	786
9	5	-14	836	831	9	8	5	549	515	10	1	0	555	545	10	5	0	424	406	11	0	-11	495	504
9	5	-12	1010	1047	9	9	-12	605	586	10	1	1	481	425	10	5	1	582	576	11	0	-9	931	903
9	5	-11	529	585	9	9	-10	608	597	10	1	3	597	538	10	5	3	803	808	11	0	-7	522	472
9	5	-10	593	549	9	9	-8	816	777	10	1	4	642	606	10	5	5	469	446	11	0	-5	733	778
9	5	-8	925	932	9	9	-6	554	511	10	1	7	570	501	10	6	-13	754	745	11	0	-3	828	856
9	5	-6	538	528	9	9	-4	1251	1226	10	2	-13	1043	1127	10	6	-10	471	476	11	0	-1	365	312
9	5	-5	661	643	9	9	0	1186	1187	10	2	-9	1224	1224	10	6	-9	825	787	11	0	1	831	765
9	5	-4	1058	1055	9	9	4	985	934	10	2	-7	655	639	10	6	-7	450	424	11	0	3	733	658
9	5	-2	441	460	9	10	-10	675	661	10	2	-5	921	923	10	6	-6	458	422	11	1	-12	648	658
9	5	0	957	953	9	10	-9	747	719	10	2	-3	590	627	10	6	-5	927	896	11	1	-11	581	588
9	5	4	853	831	9	10	-6	546	519	10	2	-1	359	336	10	6	-4	440	444	11	1	-9	856	827
9	6	-14	737	757	9	10	-5	684	681	10	2	1	748	723	10	6	-3	653	680	11	1	-7	446	430
9	6	-13	497	490	9	10	-4	377	378	10	2	3	779	748	10	6	-2	769	700	11	1	-4	[illegible]	[illegible]
9	6	-12	725	764	9	10	-3	527	521	10	2	5	821	751	10	6	-1	970	939	11	1	-3	468	481
9	6	-11	389	397	9	10	-2	754	760	10	3	-15	399	463	10	6	1	604	587	11	1	-1	457	445
9	6	-10	797	750	9	10	-1	721	699	10	3	-14	586	638	10	6	2	475	474	11	1	0	527	453
9	6	-9	516	512	9	10	2	659	669	10	3	-12	757	779	10	6	3	410	388	11	1	1	441	430
9	6	-8	519	558	9	10	3	451	412	10	3	-11	520	515	10	6	5	706	686	11	1	2	400	373
9	6	-6	757	724	9	11	-7	712	697	10	3	-10	452	450	10	7	-12	929	938	11	1	3	543	481
9	6	-5	659	613	9	11	-5	522	523	10	3	-9	404	397	10	7	-10	580	584	11	1	4	790	685
9	6	-2	1141	1136	9	11	-3	870	807	10	3	-8	765	720	10	7	-8	1133	1118	11	2	-14	774	802

(PYRIDINE)(CARBONYL)PHTHALOCYANATOOSMIUM(II) 10 FO AND 10 FC

H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC
11	2	-10	849	806	11	8	-9	721	713															
11	2	-6	1245	1228	11	8	-7	306	389															
11	2	-2	928	939	11	8	-6	555	550															
11	2	1	392	387	11	8	-5	517	506															
11	2	2	812	744	11	8	-3	714	725															
11	3	-12	463	447	11	8	-2	600	612															
11	3	-11	605	574	11	8	-1	402	371															
11	3	-9	565	563	11	9	-4	978	995															
11	3	-8	553	549	12	0	-10	690	849															
11	3	-7	893	845	12	0	-6	974	1041															
11	3	-6	451	428	12	0	-2	996	955															
11	3	5	822	819	12	1	-12	555	575															
11	3	-3	519	535	12	1	-11	534	571															
11	3	-1	579	579	12	1	-9	539	520															
11	3	0	438	450	12	1	-8	576	518															
11	3	3	660	596	12	1	-7	549	526															
11	4	-13	777	780	12	1	-5	631	660															
11	4	-11	391	394	12	1	-4	781	781															
11	4	-10	400	392	12	1	-3	624	598															
11	4	-9	806	771	12	1	-1	588	576															
11	4	-7	769	736	12	1	0	540	487															
11	4	-6	498	471	12	2	-11	455	471															
11	4	-5	984	962	12	2	-9	723	690															
11	4	-3	526	515	12	2	-7	675	589															
11	4	-1	589	608	12	2	-5	572	590															
11	4	1	510	477	12	2	-3	899	953															
11	5	-12	789	761	12	2	-1	415	406															
11	5	-8	1021	982	12	2	1	719	668															
11	5	-7	439	430	12	3	-8	815	736															
11	5	-5	380	[illegible]	12	3	-7	411	370															
11	5	-4	977	[illegible]	12	3	-4	785	804															
11	5	0	742	729	12	3	-1	534	512															
11	6	-10	902	906	12	3	0	669	661															
11	6	-9	415	436	12	4	-10	754	685															
11	6	-6	982	952	12	4	-6	950	882															
11	6	-5	435	403	12	4	-2	875	841															
11	6	-3	451	421	12	5	-9	525	476															
11	6	-2	696	632	12	5	-7	593	557															
11	6	1	349	400	12	5	-5	747	705															
11	6	2	620	588	12	5	-1	751	735															
11	7	-11	960	876	12	6	-7	549	517															
11	7	-9	562	531	12	6	-5	631	605															
11	7	-7	844	845	12	6	-3	519	487															
11	7	-5	565	546																				
11	7	-3	631	678																				
11	7	-1	790	777																				
11	7	1	435	427																				